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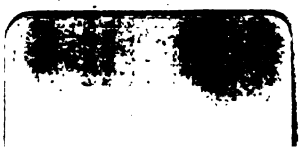

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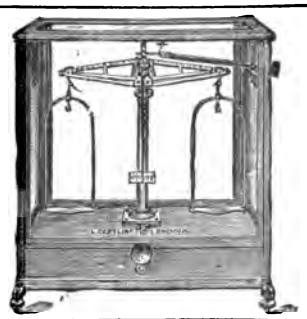

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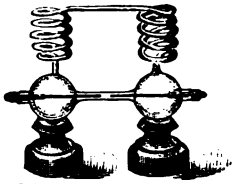

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No. 2093.—JANUARY 5, 1900.

THE RELATION BETWEEN BOILING-POINT AND MELTING-POINT IN THE HYDROCARBONS.

By THOMAS BAYLEY, Assoc.R.C.Sc.

THE accompanying table shows the melting- and boiling-points of the hydrocarbons of the paraffin, olefine, and acetylene series in absolute degrees centigrade.

The melting-point and the boiling-point *in vacuo* are points that mark two important changes in the condition of a body under the influence of heat. The melting-point is the temperature at which the internal attractions conditioning solidity are overcome. The boiling-point *in vacuo* is the temperature at which disappear the internal attractions which have, between the melting-point and the boiling-point, preserved the substance in the liquid form. There are, of course, no determinations of boiling-point made in an absolute vacuum, but some of the data in the table are for a pressure of 15 m.m., at which the external force of atmospheric pressure is reduced to an insignificant amount.

According to the usual definition, the boiling-point of a substance is the temperature at which the pressure of its vapour is equal to the pressure of the surrounding atmosphere, and since probably every substance has a vapour pressure, however small, at and below the melting-point, it would seem to follow that every liquid must boil at any temperature in an absolute vacuum if we neglect such considerations as the cohesion between the substance and the walls of the vessel, the pressure of the liquid column, &c., &c.

According to this view, the term, boiling-point *in vacuo*, has no definite meaning. At the same time the term has a very definite meaning at those low pressures which are the nearest approach to true vacua possible in ordinary chemical investigation.

If we plot the boiling-points of a liquid under various pressures the curve indicates that *in such ordinary vacua* the relations between the increment of vapour pressure and the increment of temperature undergo an abrupt change at or near the boiling-point *in vacuo* (meaning by this a very low pressure). Below this boiling-point the increment of vapour pressure is relatively small, and the heat entering the liquid is for the most part expended in raising the temperature. At this boiling-point the internal attractions having given way before the intensity of thermal vibration, the added heat no longer causes rise of temperature, but wholly passes into the latent form.

If now we suppose a liquid *in absolute vacuo* (supposed to be permanently maintained) to be placed near a source of heat, then, according to the view previously stated, it will boil at any temperature, because at that temperature it has a vapour pressure. But while the heat is passing into the substance at temperatures near to the melting-point, the thermal energy for the most part expends itself in increasing the thermal vibrations of the particles of the substance (*i.e.*, the temperature), but the ratio between

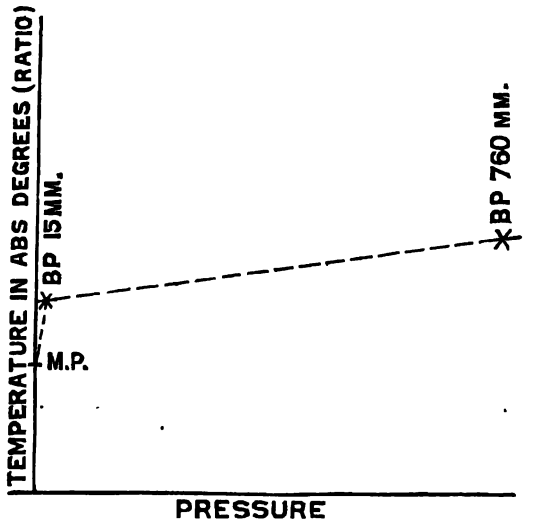


DIAGRAM SHOWING TWO POINTS ON THE CURVE OF BOILING-POINTS AT VARIOUS PRESSURES FOR $C_{19}H_{40}$ AND $C_{20}H_{42}$.

the heat so engaged and the heat rendered latent in forming vapour tends constantly to decrease, through increase of the lower term, until at length ebullition occurs at a temperature *no longing tending to rise*. This temperature may be called the "intrinsic boiling-point" of the substance. When heat is imparted to a solid, having a vapour pressure, a similar ratio exists and similarly tends to diminish, and the only difference between the two cases is that in the one case the vapour is evolved at the surface of the body (sublimation), and in the other is formed in bubbles at the region where the

Paraffins.					*Olefines (normal).*				
	M.-p.	B.-p.	Ratio: B.-p./M.-p. 760 m.m.	Ratio: B.-p./M.-p. 15 m.m.		M.-p.	B.-p.	Ratio: B.-p./M.-p. 760 m.m.	Ratio: B.-p./M.-p. 15 m.m.
CH_4	87	109	1·25						
C_2H_6			(1·5)?		C_2H_4	113	170	1·5	
C_5H_{12} (tert.)	253	282	1·11						
Normal Hydrocarbons—									
C_9H_{20}.. ..	222	423	1·9						
$C_{10}H_{22}$ ..	241	446	1·85						
$C_{11}H_{24}$ ..	247	468	1·9						
$C_{12}H_{26}$ ..	261	487	1·88		$C_{12}H_{24}$ ..	242	369†		1·52
$C_{13}H_{28}$ ..	267	507	1·9						
$C_{14}H_{30}$ ..	278	526	1·9		$C_{14}H_{28}$ ..	261	400†		1·53
$C_{15}H_{32}$ ..	283	544	1·9						
$C_{16}H_{34}$ ..	291	561	1·9		$C_{16}H_{32}$ ..	277	547 / 428†	1·98	1·54
$C_{17}H_{36}$ ..	296	576	1·95						
$C_{18}H_{38}$ ..	301	590	1·96		$C_{18}H_{36}$ ..	291	452†		1·55
$C_{19}H_{40}$ ..	305	603	2·0						
$C_{20}H_{42}$ ..	310	478*		1·54					
$C_{21}H_{44}$ ..	313	488*		1·57					
$C_{22}H_{46}$ ..	317	498*		1·57	*Acetylenes (normal).*				
$C_{23}H_{48}$ ..	321	507*		1·55					
$C_{24}H_{50}$ ..	324	516*		1·53	$C_{12}H_{22}$ ..	264	378†		1·44
					$C_{14}H_{26}$ ..	279	407†		1·46
$C_{27}H_{56}$ ..	333	543*		1·63					
$C_{31}H_{64}$ ..	341	575*		1·68	$C_{16}H_{30}$ ..	293	433†		1·47
$C_{32}H_{66}$ ..	343	583*		1·70					
$C_{35}H_{72}$ ..	348	604*		1·73	$C_{18}H_{34}$ ..	303	457†		1·50

* Beyond $C_{20}H_{42}$ the determinations of boiling-point are made at 15 m.m. pressure owing to the instability of the hydrocarbons at high temperatures and pressures.
† Boiling-point under 15 m.m. pressure.

heat gains access to the liquid. The idea of fixity is implied in the conception of boiling-point, as this term is used by chemists in describing the properties of substances, and it would be well to state the condition in the definition of the phenomenon. Whether this view of the nature of ebullition *in vacuo* be sound or unsound, it is clear that when a body boils under a small and negligible external pressure, the relation between the two crises at which temperature remains constant owing to the unloosing of internal attractions must be worthy of study.

In this communication the value of the ratio between boiling-point at or about 760 m.m. or at 15 m.m., and melting-point, has been taken for the purpose (because these are usually the only available data); this is b.-p./m.-p., but it is clear that the results might also be expressed in the form b.-p.−m.-p./m.-p. It will be observed that the ratio (b.-p./m.-p.=R) varies in the paraffin series from 1·25 for CH_4, to 2·0 for $C_{19}H_{40}$. Beyond C_{19} . . . the ratio (R-15 m.m.) is given for b.-p. under 15 m.m. of atmospheric pressure, for C_{20} . . . the value is 1·54, rising to 1·73 for C_{35} . . . So far the hydrocarbons mentioned are of *normal* constitution.

An examination of the normal olefines shows a similar state of things to exist. Ethylene, C_2H_4, the lowest member of this series, has the ratio 1 5, and from C_{12} . . . to C_{18} . . . the value of R-15 m.m. rises very little, only from 1·52 to 1·55. The variation from C_9 . . . to C_{19} in the normal paraffin series is only from 1·9 to 2·0. The ratio R for the middle portions of both series is therefore almost constant, with a tendency to increase with the increasing length and consequent decreasing stability of the chain. It is apparent that the rational values are approximately equal for corresponding members of the two series, since Meyer Wilderman (*Ber.*, xxiii., 1254) has shown that in organic series the ratio—

$$\frac{\text{Boiling-point at small pressure}}{\text{Boiling-point at 760 m.m.}}$$

is a constant. It is probable that the ratio for C_2H_6, which is not known, is 1·5 (= R for C_2H_4).

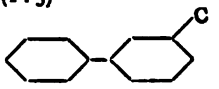
Marsh gas (CH_4), which is the most stable member of the paraffins, not being decomposed at a low red heat, has the ratio 1·25; from this to the two carbon chain in C_2H_4 the rise is rapid, viz., to 1·5. As is well known, both melting-point and boiling-point in these compounds increase with lengthening chain, the values of R indicate that boiling-point rises relatively more than melting-point.

The (normal) paraffins afford an example of a series of compounds formed by the consecutive addition of a common constituent, the group CH_2. The value of R in this series is 1·25 for the lowest member, and at the middle of the series it is almost equal for neighbouring members, but a little higher for the greater molecule. The ratio for H, the lowest member of group I., is 1·25 (from Dewar's results), and in the middle of the series Na and K have a rational value of 2·75 and 2·80 (Perman's data). The parallel is interesting.

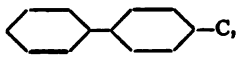
The *tertiary* paraffin C_5H_{12}, with a symmetrical molecule containing a central carbon atom directly united to four others, has the low rational value 1·11. Here, linkage of carbon to carbon causes the melting-point to be relatively high and the boiling-point to be relatively low. The hydrocarbon benzene, C_6H_6, in which the carbon chain has no ends, but is closed, and the molecule can therefore exhibit symmetry about three lines drawn through its carbon atoms and the centre of the hexagon (plane formula), has R = 1·26. Here there is no side chaining to explain the low value, but there is linkage of carbon to carbon to a greater or less extent, according to the various conceptions of the nature of the benzene ring. Kekulé's formula shows only bonding of each carbon atom to two others, with alternate double and single linkage. But this arrangement in the olefines does not co-exist with low ratio. There are several other formulæ for benzene; that of Claus and Körner and that of Ladenburg indicate a linkage of each carbon atom to three others; in Dewar's formula two of the six carbon atoms are triply united by a *para* bonding.

Just as the value of R rises from 1·25 in CH_4 to 1·5 in C_2H_4, so it is raised by the union of two rings in diphenyl from 1 26 to 1·54 (difference = 0·27).

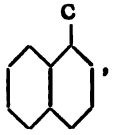
Where the two rings, as it were, partially coalesce in naphthalene, R = 1·40, which is the mean between 1·26 and 1·54. For the highly symmetrical triple ring of anthracene, with high linkage of carbon to carbon, and a central *para* bonding, the ratio is 1·28, while it is 1·63 for the isomeric but less symmetrical phenanthrene which has no *para* bonding. The effect of symmetry upon the rational value is illustrated by a study of the methyl-benzenes. $C_6H_5.CH_3$ has $R > 1{\cdot}5$. For the xylenes $C_6H_4(CH_3)_2$, the value of R is 1·42 for the symmetrical (*para*) isomer and 1·69 for the *o*-compound (difference = 0·27). In symmetrical tetra-methyl benzene (1 : 2 : 4 : 5) (durene) the ratio is 1·31, in iso-durene (1 : 3 : 4 : 5) it is $>$ 1·31, and it is 1·77 in the one-sided (vicinal) prehnitene. When the nucleus is completely encircled with six methyls in hexamethyl benzene R is 1·23, but in penta-methyl benzene, with a gap in the sentinel methyl groups, the ratio is 1·55. The ethyl-benzenes are analogous as regards the value of R, but the values are individually greater owing to the effect of the longer side chains. In methyl diphenyl (1 : 3)—



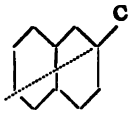
R is $> 2{\cdot}1$; in the *para* isomer,—



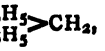
it is 1·98. Again, (*α*) methyl naphthalene,—



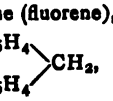
has $R > 2$; but the (*β*) isomer, which has a kind of symmetry, thus—



exhibits the lower ratio 1·68. In symmetrical diphenyl-ethylene R is 1·46, it is much higher in the unsymmetrical isomer. The ratio for symmetrical diphenylethane is 1·69, it is greater for the unsymmetrical isomer; the symmetrical and unsymmetrical diphenyl ethylenes have a similar relationship. *Para* diphenyl benzene has the ratio R = *c* 1·40, its isomer shows *c* 1·80. The effect of dissolving the *para* linkage is shown in the hydrogenising of anthracene. R for anthracene is 1·28, for the dihydride it is 1·54 (difference = 0·26). These two hydrogens are taken on at the expense of the *para* linkage. The further addition of four hydrogen atoms in the hexa-hydride raises the value of R to 1·67, *i.e.*, only by half as much, viz., 0·13. The change from naphthalene to the dihydride is from 1·40 to 1·68, the difference being 0·28. The comparison of the values of R in diphenyl methane,



and diphenylene methane (fluorene),—



is interesting. The value of R in the former is 1·799, and in the latter 1·49, and the difference is 0·31 (the boiling-points are stated approximately). Here, assuming 0·065 for the increase in R caused by the two additional hydrogen atoms (*i.e.*, the values indicated by anthracene hexahydride) added *to the rings*, we obtain a value of about 0·25 for the direct linkage between the rings, which is very near the value of the linkage difference between CH_4 and C_2H_4 (0·25) and between benzene and diphenyl (0·27). The values of R for acenaphthene, $C_{10}H_6.C_2H_4$, and for acenaphthylene, $C_{10}H_6.C_2H_2$, are the same, viz., 1·48. Here the two hydrogen atoms are added to *the fatty chain* at the expense of a double bond, and we have seen in the paraffins and olefines that no appreciable difference of rational value exists between corresponding members of these series. Taking the rational values quoted previously, at least eleven pairs of compounds can be found with differences varying from 0·26 to 0 29 (mean 0·278), while seven pairs exhibit a difference ranging from 0·12 to 0·14 (mean 0·13), and six pairs have a difference varying from 0·41 to 0·43 (mean 0·423). It must be remembered that many of the boiling-points used in the computation of the values of R in this communication are made at various pressures in the neighbourhood only of the normal pressure and at pressures frequently not stated. To the uncertainty arising in this way must of course be added that due to errors in the melting-point determinations. But as far as one can judge from the available data, limited owing to the non-existence of both melting-point and boiling-point determinations for the same compound, it would appear that the study of the ratio between melting-point and boiling-point in the hydrocarbon series throws fresh light upon the connection between molecular constitution and these physical properties of substances. In this communication an attempt has been made to discover the nature of the connection.

ON THE THEORY OF THE ACTION OF CARBONIC OXIDE ON DISSOLVED FERROCYANIDE OF POTASSIUM.

By J. A. MULLER.

On heating a solution of the yellow prussiate of potash in an atmosphere of carbonic oxide, I have already shown (*Comptes Rendus*, vol. lxxvii., p. 123), that a carbonyl-ferrocyanide of potassium is formed, and further, I proved that the reaction takes place according to the equation—

$$1.\ K_4Fe\overline{C}N_6 + CO + 2H_2O = K_3Fe\overline{C}N_5CO + NH_3 + HCO_2K.$$

However, by working under better conditions of temperature, &c. (130—135°), the coefficient of the reaction hardly exceeds 0·9, or 90 per cent, even when using a quantity of carbonic oxide slightly in excess of the amount required theoretically. If the proportion of carbonic oxide is increased the coefficient of the reaction increases. Thus, by heating for forty-eight or fifty hours to 130—135° in a Wiesnegg apparatus, fitted with an Arsonval regulator, a practically constant volume of carbonic oxide in the presence of decreasing quantities of dissolved prussiate, I obtained the following results:—

Proportion of CO_2 per molecule of prussiate.	Coefficient of the reaction.
1·3	0·91
1·7	0·96
3·2	0·98

The increase of the coefficient of the reaction with the proportion of carbonic oxide used might cause us to believe that this reaction is limited by the inverse reaction; this, however, is not the case, or at least if there is any inverse reaction, it plays only a secondary part in the limitation. This is proved by the following experiments:

A mixture composed of—

$K_3Fe\overline{C}N_5CO$	1·576 grms.
NH_3	0·081 ,,
HCO_2K	0·400 ,,
Water..	10 c.c.

is heated for fifty hours at 130—135° in an atmosphere of

nitrogen; thus, the mixture contains the first three compounds in the proportion of the inverse reaction of equation (1). On opening the tube, I found that the quantity of ammonia had remained the same, but that nevertheless a quantity of ferrocyanide of potassium, equal to 0·101 grm., had been formed, while the greater part of the carbonylferrocyanide, that is to say, 1·418 grms., had not undergone any decomposition.

In a second experiment, a solution of 1·576 grms. of pure carbonylferrocyanide of potassium in 10 c.c. of water was heated under the same conditions, that is to say, for fifty hours, to 130—135° in an atmosphere of nitrogen. The gaseous volume of the tube was equal to 79·5 c.c. at 25° and 756 m.m. pressure. On opening the tube, I first observed that this gas, in the dry state, was composed as follows:—

Carbonic oxide	6·7
Nitrogen (by difference)	93·3
	100·0

As to the liquid, it held in suspension, as also did that in the previous experiment, a very slight greenish white precipitate containing iron. It consisted of 0·072 grm. of ordinary prussiate and 0·0028 grm. of ammonia; finally, there remained 1·459 grms. of carbonylferrocyanide of potassium unchanged.

Admitting that the prussiate found in this last experiment is formed according to the following equation—

$$2.\ 4K_3Fe\overline{C}N_5CO+4H_2O=$$
$$=3K_4Fe\overline{C}N_6+4CO+(HCO_2)_2Fe+2NH_3,$$

there should be formed 0·0073 grm. of carbonic oxide (found 0·0059 grm.), and 0·0022 grm. of ammonia (found 0·0028 grm.). Considering the small quantities of the different compounds used, these figures may be looked upon as sufficiently concordant for us to infer that the ferrocyanide of potassium found in this experiment is actually formed according to the equation (2), and that it is entirely, or at any rate principally, this reaction that limits reaction (1) in the preparation of carbonylferrocyanide of potassium.

If, in the preceding experiment we replace the atmosphere of nitrogen by an atmosphere of carbonic oxide, the quantity of carbonylferrocyanide which is decomposed is almost nil (0·32 per cent). Under these conditions, therefore, the reaction (2) does not take place. The result is that by heating a solution of prussiate in the presence of a sufficient excess of carbonic oxide this prussiate should be practically entirely changed into carbonylferrocyanide; it is this that the direct experiments mentioned at the commencement of this paper have already shown.

The last experiment with which we have to deal proves that the resistance of carbonylferrocyanide of potassium to direct hydrolysis is greater, even much greater, at 130—135° than that of ferrocyanide of potassium. In fact, on heating 30 c.c. of a solution of prussiate, at 200 grms. of crystallised salt per litre, for forty-eight hours at 130—135° (the gaseous volume of the tube was then only 8 c.c., and was filled with air), there is formed 0·1380 grm. of formate of potassium, and 0·0284 grm. of ammonia. Both these compounds arise from the hydrolysis of 0·1075 grm. of CNK, which would give theoretically 0·1388 grm. of HCO_2K, and 0·0282 grm. of NH_3. During the heating, there is also formed a greenish-yellow precipitate weighing, when dry, 0·013 grm., and appearing to consist of a complex cyanide of iron and potassium.*

It is thus, to the slight stability of the dissolved ferrocyanide, compared with that of the carbonylferrocyanide, in the presence of an excess of carbonic oxide, and at a temperature not exceeding 130—135°, that we are led to attribute the formation of this latter to the detriment of the former; in the presence of water a group, CNK of the molecule of prussiate, is hydrolysed; this group, CNK, is then immediately substituted by a molecule of oxide of carbon with the formation of carbonylferrocyanide of potassium.

In all cases the presence of free carbonic oxide is necessary for the formation of this salt. I have made a considerable number of experiments in which carbonylferrocyanide ought theoretically to have been formed, without ever finding a trace of this compound when the mixture was not in the presence of carbonic oxide. On the other hand, water is no less indispensable to the reaction. In fact, by heating dry finely powdered ferrocyanide of potassium for fifty hours at 127—136°, spread in a thin layer in a sealed tube containing dry carbonic oxide (1·1 molecule), there is not a trace of carbonylferrocyanide of potassium formed.—*Bull. Soc. Chim.*, Series 3, vol. xxi., No. 10.

* When treated with fuming hydrochloric acid, a part of this precipitate is dissolved; on then diluting the mixture with water a fairly abundant precipitate of prussian blue is formed.

FLUORINE IN THE MINERAL WATERS OF SPAIN AND PORTUGAL.

By A. J. FERREIRA DA SILVA and ALBERTO D'AGUIAR.

Under this title we published, on the 1st of May this year (1899), in a Portuguese medical journal (*A Medecina Moderna*, May, 1899, p. 280—281), a preliminary note which we think is worth reproducing here with a few additions and corrections, as it appears to us to throw some light on the issue raised in the *Comptes Rendus* of the same date, by M. F. Parmentier (*Comptes Rendus*, No. 18, May 1, 1899, p. 1100—1101), in his paper, "On the Fluorine supposed to be contained in certain Mineral Waters." The following is what we then wrote:—

"Fluorine was first detected and estimated by Berzelius in the celebrated Karlsbad waters; other chemists found it later on in several different springs; the work of Nicklès on the fluorine in the waters of Plombières may be specially mentioned.

"It is, however, certain that, according to all published analyses, fluorine occurs only in traces, or in very small quantities.

"Among our own mineral waters, that of Gerez is remarkable for the quantity of this element present according to the analysis of M. Souza Reis (1885). M. Ricardo Jorge, who has examined these waters in a most searching manner, classifies them as being the most fluorised waters in Europe, and he remarks that the water from Gerez contains four times as much fluorine as that from Karlsbad (Richardo Jorge, *Caldas do Gerez, O Gerez Thermal*; Porto, 1891, p. 52).

"The illustrious professor has not only tabulated, from this point of view, the values of the other similar springs in Europe as compared with that of Gerez, but he has also shown by experimental research ("Trabalhas experimentaes e clinicas sobre as fluoretos alkalinos"; *e Caldas do Gerez*; Porto, 1888), the therapeutic action of the alkaline fluorides; and he has thus cleared up the mystery which enveloped the salutary action of these waters.

"One of us, being engaged in the analysis of the spring at Campilho at Vidago, found in this water the presence of fluorine in much greater quantity than it had been found in other springs from the northern part of Portugal; and we were even able to estimate it, an estimation which we hope to repeat by means of a new and more precise method.

"The following are the quantities of fluorine found in the two waters:—

Fluorised Portuguese Waters.

Gerez (fluoride of sodium)	0·022880 grm.
Vidago (Campilho spring) fluoride, of sodium..	0·000942 ,,

"In Spain the question of the existence of fluorine in mineral waters was examined by Dr. Joseph Casares Gil, ten years after the work done in Portugal, in a memoir presented to the Royal Academy of Arts and Science at Barcelona, and in a note dated April 16, 1896, and published in the *Boletin* of the same academy in the month of April, 1898, pp. 420—424.

"On analysing two samples of sulphurous waters, the one hot and the other cold, from the province of Lugo, in the town of the same name, at Guitiriz, Dr. Casares Gil found the following results:—

Spanish Fluorised Waters.

Lugo (fluoride of sodium)	0·0242 grm.
Guitiriz (fluoride of sodium) ..	0·0234 „

"The same chemist was of the opinion, and with good reason, that the proportion of fluorine in these two samples being so considerable, this fluorine might be detected without having recourse, as is usual, to the evaporation of several litres; and for this purpose he devised an apparatus by which the presence of fluorine is detected by the transformation of the fluoride of silicon, which can be made to contain the whole of fluorine in the water, into gelatinous silica,* he was able to obtain a very distinct silica ring when working on only 500 c.c. of these waters.

"The author quoted asserts that fluorine is to be found in many mineral waters in much higher proportions than has been observed up to the present, and that for this reason many analyses should be revised; it can be safely affirmed that fluorine is present in noticeable quantities in many alkaline and sulphurous springs in Galicia (Verin, Molgas, Burgas de Orense, Carbalho, Carballino, Caldellas de Fuy), Caldas de Reyes, Catoira, and Contrexville in France.

"How does it happen that up to the present time the presence of fluorine in these waters has never been detected?

"The author replies that very often it has not been tested for, because its presence was considered to be so rare and of such little interest; on other occasions it has not been detected on account of the inexact methods of detection which, through a typographical error in the older editions of Fresenius's "Qualitative Analysis," both in German and in the French translations, recommended the detection of fluorine by the corrosion of glass by the hydrofluoric acid, given off when the residue from the evaporation of the water (after treatment with water and precipitation by chloride of lime) was treated with sulphuric acid.

"It is, however, certain that with mineral waters containing silica and silicates, the precipitate of fluoride of calcium is found to be associated with silicate of lime, and in the treatment with concentrated sulphuric acid it is not usually hydrofluoric acid that is given off, although fluorides may, all the same, be present in the water, but fluoride of silicon, the action of which on glass is barely noticeable. In the case of siliceous waters the glass-corrosion test by hydrofluoric acid should not be used for testing for fluorine, but the transformation into fluoride of silicon.†

"The existence of fluorine in mineral waters brings up a certain number of analytical questions.

"One of these is, the exact determination of the silica in the presence of fluorine. The other is, the loss of fluorine and silica, in the form of fluoride of silica, which should take place when the sulphated residue of the waters is determined as a check on the analysis, from which it results that the residue found might be less than that calculated, although the analysis may have been conducted with the utmost care.

"It seemed to us to be of interest to verify the presence of the silica ring according to Dr. Casares Gil's method in the Portuguese fluorised waters, and in those of Caldas da Saude (at Santo Thyrso), recently analysed by one of us.

"We verified the accuracy and the sensitiveness of the process, and, that of the three Portuguese mineral waters, those of Gerez gave a perceptible reaction with only 250 c.c., and those of Campilho with 500 c.c.

"In the same quantities, fluorine was not detected in the waters of Caldas da Saude.

"We propose carrying on this research on mineral hydrology, which appears to be of great interest."—*Bull. Soc. Chim.*, Series 3, vol. xxi., No. 20—21.

* Dr. Casares Gil describes his apparatus in a note sent to M. R. Fresenius, and published in the *Zeit. für analytische Chemie*, vol. xxiv., p. 546.

† The error to which M. Casares Gil makes allusion is still found in the 7th French edition of 1885 ("Traité d'Analyse Qualitative," de R. Fresenius, p. 386), where we find quoted the reaction 146·5 of page 241, when it should have been 146·6. In the 8th edition of 1896 (pp. 254 and 429), and in the 9th edition of 1897 (pp. 302 and 494) the error has been corrected.

ON THE

ANALYSIS OF VULCANISED INDIARUBBER.

By CARL OTTO WEBER.

The method I proposed some time ago (*Chemiker Zeitung*, 1894, 1696) for separating indiarubber from the mineral matter it contains, a method which consists in boiling the finely divided rubber in a flask fitted with a vertical condenser with nitrobenzene, has given in practice very satisfactory results. The only drawback to this method is the difficulty sometimes experienced in eliminating the mineral matter from the solution of the products of decomposition of the indiarubber.

In most cases this inconvenience can be guarded against by diluting the solution with a large quantity of ether. If we have to deal with African rubbers, the quantities of ether to be used are relatively very large. Further, the treatment with nitrobenzene gives rise to a phenomenon that I have only recently observed for the first time.

While analysing a sample of rubber (*a*) by the viscous decomposition method, and another sample (*b*) made in the same manner and of irreproachable quality, I obtained the following results:—

	a. Per cent.		*b.* Per cent.	
Oily extract	8·35		2·82	
Indiarubber	9·85		12·73	
Inorganic matter ..	79·9	63·11 min. 16·79 carb.	82·34	62·81 min. 19·53 carb.
Sulphur (total) ..	1·89		2·10	

The presence of such considerable quantities of carbon or carbonaceous material, on the one hand, and the incredibly small proportion of rubber in the product, on the other hand, has caused some apprehension as to the correctness of this analysis, and further examination has only strengthened this doubt.

Under ordinary conditions the figures representing "oily extract" would be attributed to the rubber from which this extract was made, seeing that, as far as the manufacturer is concerned, the extract forms a part of the rubber itself. But in these two samples the quantity of extract is nearly triple the amount that it is in ordinary indiarubber. Although the proportion of extract varies considerably with the kind of rubber, still, so pronounced a difference as that observed between the samples *a* and *b* appears to be hardly credible. Further, in the samples analysed, the increase in the quantity of extract found is counteracted by the diminution of the amount of rubber found, which seems to indicate that the oily extract is for the most part only a product of decomposition of the indiarubber. The analysis also shows that the proportions of carbon follow the proportions of rubber present. All these facts tend to show that the carbon found did not pre-exist in the product, but results from the action of

the boiling nitrobenzene on the indiarubber. It is also possible that its formation is due to the action of the high temperature at which nitrobenzene boils.

I endeavoured to dissolve the samples in question in nitrobenzene at a temperature of 170—190°, but the attempts were fruitless. Either the solution was incomplete, or else the carbon made its appearance in the same proportion as in the analysis quoted above.

Starting from the idea that the preliminary swelling of the rubber in chloroform might facilitate its disaggregation by the boiling nitrobenzene, I placed 3 grms. of each sample into the wide-necked flasks used for the treatment with nitrobenzene; I then added 3 c.c. of chloroform to each, corked the flasks, and allowed them to stand for about an hour. At the end of this time the indiarubber was enormously swollen. Without driving off the chloroform I added to each flask 50 c.c. of nitrobenzene, fitted a vertical condenser to each flask, and heated to boiling in a paraffin bath. At the end of an hour the reaction was stopped, and after cooling, 100 c.c. of ether were added to each flask, and the contents then filtered. The results obtained were as follows :—

	a. Per cent.	*b.* Per cent.
Inorganic material.. ..	63·33	63·02

There was not a trace of carbon present. From these results it appears that the previous swelling of the rubber by means of chloroform has the effect of totally preventing carbonisation. The favourable action exercised by the chloroform in the process is beyond doubt; but it must not be forgotten that the addition of chloroform to the nitrobenzene considerably lowers the boiling-point of the latter. A direct experiment has shown that a mixture of chloroform and nitrobenzene in the proportions mentioned above boils at a temperature of from 170 to 180°. It is to the action of the chloroform on the rubber at this temperature that I attribute the energetic disaggregation of the latter.

The experiment described above appears also to show that the carbonisation of the rubber by boiling nitrobenzene was principally due to the influence of the high temperature, which influence was probably seconded by the oxidising action of the nitrobenzene. But the exact analysis of the organic matters obtained in the two series of analyses has shown where to look for the true cause of the carbonisation of the rubber.

Treatment by Nitrobenzene only.

	a. Per cent.	*b.* Per cent.
Carbonaceous matter	17·22	19·93
Mineral matter—		
$CaCO_3$	20·78	21·04
$PbCO_3$, PbO	40·27	41·83
PbS, $PbSO_4$	2·10	1·00
Total mineral matter	63·15	63·87

Treatment by the Mixture of Chloroform and Nitrobenzene.

	a. Per cent.	*b.* Per cent.
Carbonaceous matter	traces	traces
Mineral matter—		
$CaCO_3$	21·02	21·11
$PbCO_3$, PbO	32·22	31·56
PbO_2	9·97	10·05
PbS, $PbSO_4$	1·05	0·88
Total mineral matter	64·26	63·60

It is noticeable that the mineral matter isolated in treating the rubber with the mixture of chloroform and nitrobenzene contains a considerable quantity of peroxide of lead, while at the same time it is almost completely free from carbon. On the contrary, the treatment of rubber with nitrobenzene at once gives a large proportion of carbon and hardly any peroxide of lead. It is thus beyond doubt that at the temperature of boiling nitrobenzene (208°) the peroxide of lead contained in the rubber attacks the latter with the formation of carbon, a reaction which, at the temperature of the boiling mixture of chloroform and nitrobenzene, does not evidently take place. It is hardly probable that the peroxide of lead found exists as such in the inorganic residue.

It seems, moreover, certain that it comes from the minium during the treatment of the residue by dilute nitric acid. Minium is often used in the manufacture of vulcanised articles by heat; above all, if a soft rubber is used. In such a case the minium exercises a hardening action, that is to say, slightly oxidising (?). By calculating the peroxide of lead found in the second analysis as minium, the composition of the inorganic residue should be expressed by the following figures :—

	a. Per cent.	*b.* Per cent.
$CaCO_3$	21·02	21·11
$PbCO_3$, PbO	22·92	22·19
Pb_2O_3	19·27	19·42
PbS, $PbSO_4$	1·05	0·88
	64·26	63·60

These figures show that the rubber used contained almost equal quantities of carbonate of lime, carbonate of lead, and minium.

From the purely chemical point of view, the question of deciding what transformation is undergone by the rubber under the action of the treatment with nitrobenzene alone, or in the presence of chloroform, is one of great interest; but up to the present I have not had an opportunity of studying this question. As I have already stated, the indiarubber does not exist as such when in nitrobenzene solution. It must therefore be, that the treatment has the result of degrading the molecule of indiarubber.

Now, we know that when submitted to dry distillation rubber is transformed almost quantitatively into hemiterpene, dipentene, and polyterpenes. We might therefore suppose that vulcanised rubber would undergo an analogous decomposition when treated with boiling nitrobenzene. To this it might be objected that non-vulcanised rubber becomes quite viscous at the temperature of boiling nitrobenzene, without giving any products of distillation, while vulcanised rubber is hardly changed at this temperature.

But still it is always the fact that indiarubber commences to decompose, though slowly, in the neighbourhood of 200°. It is again very probable, considering the nature of the products of decomposition of indiarubber, that isoprene, which constitutes about 15 per cent of the portion distilled, is not an immediate product of the decomposition of rubber, and that there are first formed bodies of a high molecular weight (polyterpenes ?), which then give birth to citrene and isoprene. This view seems to be confirmed by the fact that I have often noticed in the dry distillation of different kinds of rubber, that the slower the distillation, and therefore the lower the temperature, the lower is the return of citrene and isoprene. The best results are obtained by introducing small portions of rubber at a time into a retort heated to redness. From these facts I think I may conclude that, under the influence of a high temperature, indiarubber does not decompose into small molecules, but first gives rise to products of a high molecular weight.

Further, in the treatment of indiarubber by nitrobenzene—that is to say, at a relatively low temperature—rubber ought to give products of a high molecular weight,

perhaps even one single product. If such is the case, we should be able not only to estimate rubber directly in manufactured articles, but, further, we should by so doing be making an important step forward towards a knowledge of the nature and the size of the molecule of india-rubber. I am continuing the experiments I have started in this direction.—*Zeitschrift fur angewandte Chemie*, 1898, p. 313.

THE ACTION OF ACETYLENE ON THE OXIDES OF COPPER.*

By F. A. GOOCH and DEFOREST BALDWIN.

In a recent paper by Erdmann and Köthner (*Zeitschr. fur Anorg. Chem.*, xviii., 49) an account is given of the formation of a peculiar, light brown, highly voluminous substance by the action of acetylene below 250° C. upon cuprous oxide, or even (though more slowly) upon copper. The product obtained by passing acetylene during eighteen hours over 1 grm. of cuprous oxide (prepared from copper sulphate, grape sugar, and sodium hydroxide) amounted to 7 grms., and filled a space of nearly 300 c.m.³. At higher temperatures a black carbonaceous mass is the result, and at red heat (400—500° C.) carbon is deposited in graphitic condition. The light brown fluffy material yielded cuprous chloride to hydrochloric acid, a distillate from its mixture with zinc dust possessing the characteristic of naphthene, or, at higher temperature and under rapid heating, aromatic compounds among which naphthalene and a kresol were indicated. Erdmann and Köthner classify this body as a very complex but non-explosive copper acetylene (acetylen-kupfer), and from their analyses deduce the formula $C_{44}H_{64}Cu_3$. Apart from the unusual constitution of this symbol, its most striking peculiarity is that it implies a loss of carbon, rather than hydrogen, from the acetylene in the reaction with cuprous oxide—a condition of affairs which would be most remarkable in the light of Campbell's experience (*Amer. Chem. Journ.*, xvii., 690), according to which acetylene, passed over palladinised copper oxide, yielded water at 225—230°, and carbon dioxide only when the temperature rose to 315—320° with the formation of a black deposit. Upon scrutinising the figures of Erdmann and Köthner with care, however, it appears that the formula given by these investigators rests upon some oversight in calculation: the ratio of carbon atoms to hydrogen atoms proves to be actually, according to the data given, 6·45 : 5·70; which means, of course, that the new product is deficient, as would be expected, in hydrogen (not in carbon) as compared with acetylene.

As to the content of the new substance in copper, the analytical data are unfortunately ambiguous; for we note the weights found of *copper oxide* converted into percentages of *copper* without preliminary reduction. If the fault is typographical and in the analytical data, the calculated percentages of copper being correct, the average percentage of copper amounts to 15·43; if, on the other hand, the analytical data are right, the error being in their reduction, the percentage of copper amounts to 12·92. In the one case the summation of the analysis leaves a deficiency of about 1·5 per cent, and in the other of about 4 per cent, which in either case may really represent oxygen in the substance. This condition of matters leaves the "acetylen-kupfer" of Erdmann and Köthner in uncertain standing.

More than thirty years ago it was noticed by Berthelot (*Ann. d. Chim. et d. Phys.* [4], ix., 448) that acetylene is polymerised by heat or decomposed partially into carbon and hydrogen, and that such action takes place more readily and at lower temperatures in presence of metallic iron with production of carbon, hydrogen, and compounds different from those formed by heat alone.

Moissan and Moureu (*Comptes Rendus*, cxxii., 1240) have observed the incandescence of acetylene passed over finely divided iron, cobalt, nickel, or platinum, at the ordinary temperature, with production of carbon, hydrogen, and pyrogenic compounds, and have found the occasion of such behaviour in the porosity of the metals employed.

It would seem natural, however, that the presence of oxygen, free or combined, may also play a considerable part in such phenomena, just as appears to be the case in the peculiar action recorded by Gruner (*Ann. d. Chim. et d. Phys.*, [4], xxvi., 5) of carbon monoxide upon iron reduced by hydrogen, which, as Moissan has shown (*Ann. d. Chim. et d. Phys.* [5], xxi., 199), is produced pure only with the greatest precaution, and generally carries a large proportion of ferrous oxide. The fact that the "acetylen-kupfer" of Erdmann and Köthner is produced more easily by the action of cuprous oxide upon acetylene than by the action of metallic copper upon acetylene, suggests that it may be the oxidising power of the cuprous oxide which gives to this reagent its peculiar activity. The question arises, therefore, as to whether the copper is in reality an essential constituent of the compound of Erdmann and Köthner.

In our experiments upon the action of acetylene upon the oxides of copper (and other elements) we have conducted the gas (made in the ordinary way by the action of water on calcium carbide, and kept over water) over the oxide contained in a porcelain boat placed within a glass tube, 2 c.m. in diameter and 50 c.m. long, which was heated over a small combustion furnace. The glass tube was fitted at each end with a rubber stopper, one carrying a smaller tube for the introduction of the acetylene and a high-temperature thermometer so held that its bulb rested horizontally immediately over the boat containing the oxide, while the other was fitted with a water-trap. In the preliminary experiments no attempt was made to purify the acetylene employed other than to keep it over water, or, since water is a product of its action upon oxides, to dry it: in later experiments, to secure products for careful analysis, it was dried and purified with care.

We found that 225° C. is the temperature most favourable for the formation of the voluminous product obtained by acting with acetylene upon cuprous oxide as described by Erdmann and Köthner. At this temperature the tube is choked rapidly with the fluffy product, and water forms, but, as Campbell found in his experiments upon palladinised copper oxide, no appreciable amount of carbon dioxide is produced. The content of the product in copper varies in the sample and in different experiments, our results lying between 1·54 per cent and 24·21 per cent of the substance taken for ignition.

It appeared, also, that the action of acetylene upon cupric oxide is precisely similar to that upon cuprous oxide excepting the evident reduction of the former oxide early in the action. The amount of copper in the product of such action varied in our experiments from 6·53 per cent to 21·30 per cent. In one case the experiment of re-submitting to the action of acetylene a product containing 9·34 per cent of copper was made with the result that a new growth of the substance formed which on analysis yielded 3·87 per cent of copper.

A roll of copper gauze carefully reduced in hydrogen and then oxidised at one end in the outer flame of a Bunsen burner, gave, when acted upon by acetylene at 225—250° C., the characteristic deposit upon the oxidised end only, the unoxidised end being merely discoloured.

These results go to show that, while metallic copper may at comparatively high temperatures induce the polymerisation of acetylene, it is an oxidising action which starts at moderately low temperatures the formation of the peculiar derivatives under consideration. Thus we find that ferric oxide heated in acetylene at

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, viii., Nov., 1899.

	Weight of substance taken. Grm.	Found. CO_2. Grm.	H_2O. Grm.	CuO. Grm.	Calculated. C. Grm.	H. Grm.	Cu. Grm.	O by difference. Grm.
1.	0·1170	0·3978	0·0673	0·0022	0·1085	0·0075	0·0018	−0·0008
2.	0·2247	0·7489	0·0979		0·2042	0·0109		
3.	0·1096	0·3678	0·0488	0·0045	0·1003	0·0054	0·0036	0·0003
4.	0·1360	0·4116	0·0579	0·0182	0·1123	0·0064	0·0146	0·0027
5.	0·1188	0·3 98	0·0461	0·0317	0·0845	0·0051	0·0253	0·0039

	1.	2.	3.	4.	5.
Per cent of carbon	92·74	90·88	91·51	82·57	71·13
„ hydrogen	6·41	4·85	4·93	4·71	4·29
„ copper	1·54		3·29	10·74	21·30
„ oxygen	—		0·27	1 98	3 28
	100 69		100·00	100·00	100·00

temperatures varying from 150° to 360°, according to circumstances, darkens, glows, and gathers with evolution of heat a dark carbonaceous deposit. In the products of such action we have found the content of iron varying from 2·80 per cent to 5·86 per cent.

Silver oxide, too, acts upon acetylene: thus, in one experiment, action was evident at the ordinary atmospheric temperature, a violent explosion, which completely shattered the boat and scattered metallic silver upon the sides of the glass tube, following before the temperature reached 100°.

In the locally violent explosion of the last experiment we have evidence of the formation in the early stage of an acetylide which is decomposed later when the temperature of dissociation is reached. In the experiments with the oxides of copper and iron, the temperature at which the acetylene begins to act is evidently above the point at which sensitive acetylides would naturally dissociate, and we have in the observed phenomena no evidence of the formation of such compounds of copper and iron under the conditions of experimentation.

In experiments 1 to 3 of the accompanying table are given the results of the analysis of several products obtained by conducting acetylene (purified by passing through a solution of mercuric chloride in hydrochloric acid and dried over caustic potash) over pure cuprous oxide. The temperature was kept in these experiments at 225°, and in the course of a half hour the tube was choked completely by material compacted by the pressure to 1, a spongy mass of light brown colour on the exterior next the walls of the tube; 2, darker within; and 3, nearly black in the bottom of the boat, where the cuprous oxide lay originally.

In experiments 4 and 5 the substances analysed represent the products of the action af acetylene (not specially purified) on cupric oxide.

The oxygen present in these products is obviously proportional to the amount of copper, and is never more than enough to be completely accounted for upon the supposition that some of the original oxide taken still holds its oxygen. So far as the analyses show, the product of lightest colour, 1, contains little copper and no oxygen; the darkest product, 3, obtained from the cuprous oxide contains oxygen corresponding to a mixture of two parts of copper with three parts of cuprous oxide; the oxygen in the products of 4 and 5, obtained by acting upon cupric oxide, is approximately enough to correspond to a mixture of cuprous and cupric oxides in equal proportions. This fact, taken in connection with the great range of variation in proportion and the minimum to which the copper falls in the product, which would be least likely to include contaminating metal or oxide, suggests very strongly the probability that the oxygen present is in union with copper, and that the copper is held mechanically as metal or oxide, and is not the essential constituent of an organic compound. Leaving out of consideration, therefore, the copper and copper oxides, and calculating the composition of the products assumed to consist essentially of carbon and hydrogen, we derive the following statement:

	1.	2.	3.	4.	5.
Per cent of carbon ..	93·54	94·93	94·88	94·60	94·31
Per cent of hydrogen	6·46	5·07	5·12	5 40	5·69
	100·00	100·00	100·00	100·00	100·00

These figures correspond to symbols varying from $C_{12}H_{10}$ to nearly $C_{16}H_{10}$, with an average approximating $C_{14}H_{10}$, the symbol of anthracene or paranthracene. The analytical data of Erdmann and Köthner point in the average to a product corresponding more nearly to the first of these symbols than to either of the others. The product is doubtless variable with the temperature and the activity of oxidation. Thus, in one experiment in which acetylene was passed over ferric oxide, the action began at 365° with incandescence, as described by Moissan and Moureu (*loc. cit.*), and the analysis of the product (carbon = 91·53) hydrogen = 1·36, Fe = 5·85, O = 1·26) indicates a proportion of carbon to hydrogen about four times as great as that of the average product of action at 225 on the oxides of copper.

Finally, we find no evidence that the product of the action of acetylene on the oxides of copper under the conditions of our experimentation is other than a mixture of a hydrocarbon or hydrocarbons with metallic copper or an oxide of copper, and, probably, in the darker preparations, some free carbon.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 21st, 1899.

Professor THORPE, F.R.S., President, in the Chair.

DR. KOHN was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Granville Reginald Gee, Falcon Villa, Howard Street, Gloucester; Alfred William Harvey, 13, Pendrell Road, St. Catherine's Park, Hatcham, S.E.; Thomas Ebenezer Mackenzie, 6, Cardonald Terrace, Cardonald, near Glasgow; William Mair, 388, Morningside Road, Edinburgh; John Livingston Rutgers Morgan, 47, Bayard Street, New Brunswick, New Jersey, U.S.A.; James Fraser Readman, 50, Jane Street, Glasgow; James McConnell Sanders, 37, Summerhill Road, Dartford; and of Sidney Calvert, Missouri State University, Columbia, Missouri, whose Certificate was approved by Council under Bye-law I. (3).

The SECRETARY read the following letter, written by the President at the request of the Council, to Professor Emil Fischer, a Foreign Member of the Society, con-

gratulating him on the completion of the twenty-fifth year of his Doctorate, together with a translation of Professor Fischer's reply.

16th November, 1899.

My dear Colleague,—As President of the Chemical Society, I have the pleasure to offer you, by request of the Council, the warmest congratulations of the Fellows on the completion of the twenty-fifth year of your Doctorate.

It is not often that so short a span of intellectual activity in the life of any one individual comprehends such a brilliant series of scientific triumphs as those with which you have enriched chemistry.

That you may long be spared to cultivate the many fields of fruitful inquiry you have so assiduously opened up is the fervent wish of this Society, which recalls with pride that it numbers you amongst the most distinguished of its Foreign Members.—I have the honour to be, with great respect, my dear Colleague, your obedient humble Servant,

T. E. Thorpe, President.

To Professor Dr. Emil Fischer, Berlin.

The following is the translation of Professor Fischer's reply:—

10, Dorotheenstrasse, Berlin,
29th November, 1899.

Highly esteemed President,—I have been most agreeably surprised by the honour as well as the heartiness of the congratulations which you have conveyed to me in the name of the Chemical Society, on the occasion of the celebration of the 25th year of my Doctorate. Your Society and other learned bodies in England have indeed done me so much honour that I am obliged to ask myself, with diffidence, whether it bears any relation to my achievements.

The time when the fundamental principles of our Science were laid down, and when it was possible for the individual investigator to stamp the impress of his own mind upon it, is long since past, and, in the gigantic structure which it now represents, each fellow worker can only finish some small fragment, or it may be, if he is fortunate, a pretty balcony or a striking turret. The progress of the whole is, however, most readily accomplished by the combined efforts of numerous gifted and skilled observers. This co-operation, so characteristic of the scientific work of the present day, is exhibited in its highest form by the great Chemical Societies, of which yours is the oldest and has served as a model for the others.

I am proud, therefore, to be allowed to belong to it as a Foreign Member, and I beg, esteemed President, that you will be so good as to convey to your Council my heartfelt thanks for their kind congratulations.—With the highest esteem, yours obediently,

Emil Fischer.

Prof. Dr. Thorpe,
President of the Chemical Society, London.

Of the following papers, those marked * were read:—

*163. "*On the Refractive and Magnetic Rotatory Powers of some Aromatic Hydrocarbons, and on the Refractive Powers of Mixtures.*" By W. H. Perkin, LL.D., F.R.S.

The first part of the paper deals with a comparison of the magnetic rotatory and refractive powers of aromatic hydrocarbons undertaken to see if these properties were analogous in character. Most of the hydrocarbons employed were of exceptional purity, many being prepared from the pure salts of their sulphonic acids. An account is given of the preparation and properties of *isobutylbenzene*, prepared by this method for the first time.

With regard to the refractive powers of these substances, the results obtained show that the replacement by methyl of a hydrogen atom in the nucleus gives a value considerably *larger* than when the replacement takes place outside, and this is so certainly up to four such replacements, and therefore probably for the six. The replacement of hydrogen outside the nucleus by methyl (as in the conversion of toluene into ethylbenzene) also gives results somewhat higher than those calculated, but these values tend to coincide more nearly as the distance from the nucleus increases.

With the magnetic rotations the reverse was found to hold, as a considerably *smaller* increase in the rotation is produced by a change in composition of CH_2 in the nucleus, than when it occurs outside. This holds for four replacements, and therefore probably for all.

These results are remarkable when the general correspondence between the magnetic rotatory and refractive powers of compounds is considered.

The second part refers to the refractive powers of mixtures. It has been shown that in the case of the magnetic rotations of mixtures these are usually less than those calculated from the molecular rotations of the constituents when their specific rotations are widely apart; hence it was of interest to determine whether a similar difference existed in the case of the refractive powers of mixtures. It was found that when the two constituents had very widely differing indices of refraction, the specific or molecular refraction was considerably lower than that calculated, but that when indices were nearly the same the calculated and observed values agreed very well. That this latter statement holds was shown by means of a mixture (*a*) of fatty compounds, (*b*) of aromatic compounds. These differences were most marked in mixtures of two fatty compounds having indices widely apart; in mixtures of a fatty with an aromatic compound they were less pronounced.

Many of Zecchini's experiments with mixtures give results of a similar character to those described above, but exceptions occur which seem to indicate that although the differences between the refractive powers of the substances employed appear to be the chief cause of these peculiar results, other causes must also be at work. A new and improved arrangement for reading the scale and vernier of the spectrometer was described and shown.

Discussion.

The President, after recalling the fact that differences similar to those observed by the author had been also observed with regard to the viscosity of pure compounds and mixtures of them, inquired why mixtures in equal volumes rather than in molecular proportions had been employed in this investigation.

Dr. Gladstone remarked that the results of the author to a certain extent explained some of the difficulties met with by early workers in the field of molecular refraction, and that many of them had observed similar small differences which were yet quite beyond the limit of experimental error. There was no doubt that researches such as those of Dr. Perkin must tend to throw more and more light on the causes of these small divergences in the optical properties and hence to reveal more fully the structure of the molecule.

Dr. Perkin, in reply, said that the use of mixtures of equal volumes of the constituents had originated in his work on magnetic rotation, as such mixtures allowed of more ready comparison of the indices of refraction, densities, &c., of the mixtures and their components.

164.* "*Formation of α- and β-Acrose from Glycollic Aldehyde.*" By Henry Jackson, B.A.

When a dilute (3 per cent) aqueous solution of glycollic aldehyde is allowed to stand with a 1 per cent solution of soda for fifteen hours at 0°, it is still able to reduce Fehling's solution in the cold (though not so strongly as a solution of glycollic aldehyde), and to restore the colour to an alcoholic solution of magenta which has been decolourised by sulphurous acid. If the condensation product be neutralised with acetic acid, then heated on the water-bath with phenylhydrazine acetate, an osazone is obtained which, on purification and fractional treatment with various solvents, can be shown to contain tetrosazone

[obtained previously by Fischer and Landsteiner (*Ber.*, 1892, xxv., 2549), by condensing glycollic aldehyde with soda at 0°], and two hexosazones, α-acrosazone m. p. 217°, and β-acrosazone, m. p., 158°.

If the condensation be allowed to proceed for a longer time at 0°, it is found that the power of reducing Fehling's solution in the cold gradually diminishes until, after standing six days, it no longer reduces in the cold, but quickly on warming. The osazone obtained consists almost entirely of a mixture of the two hexosazones (α and β-acrosazone), only a trace of tetrosazone being found.

DISCUSSION.

Mr. FENTON said that it was evident, both from these and from previous observations, that the condensation products of glycollic aldehyde and of formaldehyde were identical. It was probable that glycollic aldehyde was an intermediate stage in the condensation of formaldehyde, especially as Pechmann (*Ber.*, 1897, xxx., 2459) had shown that the osazone of glycollic aldehyde could be obtained directly from formaldehyde and phenylhydrazine in acetaldehyde solution.

165. "*The Interaction of Mercurous Nitrite and Ethyl Iodide.*" By P. C. RAY, D.Sc.

When mercurous nitrite acts upon ethyl iodide, about equal quantities of nitroethane and its isomeride, ethyl nitrite, are formed. The yield is, however, somewhat poorer than when silver nitrite is used, owing to the formation of very compact lumps of mercurous iodide, which prevents the reaction from becoming complete.

166. "*On Mercurous Iodide.*" By P. C. RAY, D.Sc.

When the residue in the flask after the interaction of mercurous nitrite and ethyl iodide is heated in a tube, mercurous iodide sublimes off between 190° and 210°. The compact mass of crystalline tablets thus obtained shows all gradations of tint from lemon- and orange-yellow to dark brown.

167. "*The Action of Alkyl Iodides on the Mercuric Iodide Sulphides of the Fatty Series.*" By SAMUEL SMILES, B.Sc.

The alkyl sulphides unite with mercuric iodide to form compounds of the general formula R_2SHgI_2. Like other addition products of the sulphides, their constitution should be represented as—

$$\begin{matrix} R \\ R \end{matrix}\!\!>\!S\!<\!\!\begin{matrix} HgI \\ I \end{matrix}.$$

The following members of the series were examined:—

Me_2SHgI_2, long, yellow needles from acetone, m.-p. 75°.
$MeEtSHgI_2$, short prisms from acetone, m.-p. 59°.
Et_2SHgI_2, yellow prisms from acetone, m.-p., 52°.
$(C_5H_{11})_2SHgI_2$ (isoamyl), yellow oil.
$(C_7H_7)_2SHgI_2$, pale yellow plates from acetone, m.-p. 37—38°.

These substances are unstable, readily splitting off mercuric iodide when exposed for any length of time to the air, or when boiled with water. Dimethylsulphide mercuric iodide, when treated with methyl iodide, yields the same product as that obtained from trimethylsulphine iodide and mercuric iodide, namely Me_3SIHgI_2. The same holds good for the triethyl derivative. Hence the double salts formed from mercuric iodide and sulphine iodide appear to contain hexavalent sulphur, as their constitution may be represented as—

$$R_3{:}S\!\!\begin{matrix} =I_2 \\ <HgI \end{matrix}.$$

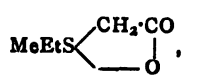
Therefore, it was hoped that an investigation of the derivatives containing different alkyl groups would yield evidence as to the stereo-chemical nature of hexavalent sulphur. So far, however, only negative results have been obtained.

The following substances were prepared and examined:

Me_3SI_2HgI, large, yellow prisms from acetone, m.-p. 165°.
From—(1) Me_3SI and HgI_2; (2) Me_2SHgI_2 and MeI.
Me_2EtSI_2HgI, long, yellow prisms from acetone, m.-p. 87°.
From—(1) Me_2EtSI and HgI_2; (2) Me_2SHgI_2 and EtI; (3) $MeEtSHgI_2$ and MeI.
$MeEt_2SI_2HgI$, bright yellow prisms from acetone, m.-p. 67°.
From—(1) Et_2MeSI and HgI_2; (2) Et_2SHgI_2 and MeI; (3) $MeEtSHgI_2$ and EtI.
Et_3SIHgI_2, bright yellow leaflets from acetone, m.-p. 112°.
From—(1) Et_3SI and HgI_2; (2) Et_2SHgI_2 and EtI.

Just as in the case of the nitrogen compounds such isomerism as might be expected here may be prevented by the presence of methyl or ethyl groups, it will therefore be necessary to repeat this with derivatives containing more complex radicles.

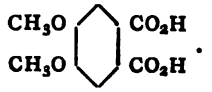
Attempts were also made to resolve methyl ethyl thetine,—



into optically active components, but without success.

168. "*On Brasilin and Hæmatoxylin.* Part III." By A. W. GILBODY and W. H. PERKIN, jun.

In a previous communication (*Proc.*, 1899, xv., 27), an acid obtained from brasilin was described which melted at 155° and gave, on analysis, numbers agreeing with the formula $C_{15}H_{16}O_6$. This acid, on oxidation with potassium permanganate, yielded a dibasic acid melting at 200—203°, which, when heated with hydrochloric acid at 160°, was decomposed with formation of catechol. Further investigation has shown that this acid, which at first was thought to have the formula $C_{10}H_{10}O_3(CO_2H)_2$, is in reality *metahemipinic acid*,



This has been conclusively proved by converting the acid into its anhydride and characteristic ethylimide (m.-p., 230°). *Tetramethylhæmatoxylin*, when oxidised with chromic acid, also yields considerable quantities of *metahemipinic acid*, a proof that hæmatoxylin, like brasilin, is a derivative of catechol. As this result does not agree with the statement of R. Meyer (*Ber.*, 1879, xii., 1393) that hæmatoxylin, on distillation, yields pyrogallol and resorcinol, we have repeated this work, and find that the products of the destructive distillation of hæmatoxylin contain considerable quantities of pyrogallol, but, as far as we could determine, no trace of resorcinol.

It follows, therefore, from these experiments, that whereas brasilin is a derivative of resorcinol and catechol, hæmatoxylin is a derivative of pyrogallol and catechol.

The formation of metahemipinic acid from both brasilin and hæmatoxylin shows that the formulæ which we suggested for these substances require some modification. It would be easy to construct formulæ which would take these new facts into account, but we prefer to wait until further experimental evidence is forthcoming before doing so. We hope, therefore, that others working in this field will allow us to reserve for a short time the deduction of any formulæ for brasilin and hæmatoxylin which are based on the new results described in this and the two preceding preliminary communications.

It should be noticed that the melting-point of tetramethylhæmatoxylone is incorrectly given as 170° in the previous communication, it should be 183—186° with vigorous decomposition.

169. "*The Action of Alcoholic Potash on Monobromoglutaric Ester.*" By N. E. BOWTELL and W. H. PERKIN, jun.

A short time since (*Trans.*, 1899, lxxv., 50) it was

shown by J. F. Thorpe and one of us that when *monobromodimethylglutaric ester*,—

$$CO_2Et{\cdot}CHBr{\cdot}CMe_2{\cdot}CH_2{\cdot}CO_2Et,$$

is digested with alcoholic potash, it yields a mixture of *cis*- and *trans*-dimethyltrimethylenedicarboxylic acids (caronic acids),—

$$\begin{array}{c} CMe_2 \\ \diagup\quad\diagdown \\ CO_2H{\cdot}CH\text{—}CH{\cdot}CO_2H. \end{array}$$

It occurred to us that it would be interesting to investigate the behaviour of *monobromoglutaric ester*,—

$$CO_2Et{\cdot}CHBr{\cdot}CH_2{\cdot}CH_2{\cdot}CO_2Et.$$

under similar conditions, as in this case there are two possibilities, namely, either glutaconic acid,—

$$CO_2H{\cdot}CH{:}CH{\cdot}CH_2{\cdot}CO_2H,$$

or trimethylenedicarboxylic acid,—

$$\begin{array}{c} CH_2 \\ \diagup\quad\diagdown \\ CO_2H{\cdot}CH\text{—}CH{\cdot}CO_2H, \end{array}$$

could be formed.

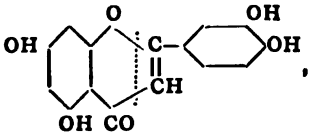
Monobromoglutaric ester was obtained as a colourless oil boiling at 165—170° (35 m.m.) by brominating glutaric anhydride and pouring the product into alcohol. When this ester is decomposed by alcoholic potash, an acid is obtained melting at 175°, which on examination was found to consist of trans-*trimethylenedicarboxylic acid*, and we were unable to isolate even traces of glutaconic acid.

This remarkable result points to the possibility that many substances produced by similar reactions and described as unsaturated may in reality be derivatives of trimethylene, tetramethylene, &c.

We are engaged in investigating the action of alcoholic potash on the esters of bromine substitution products of adipic, pimelic, and allied acids.

170. "*Luteolin*. III." By A. G. Perkin.

Previous investigations (*Trans.*, 1896, lxix., 206, 799) indicated that luteolin, $C_{15}H_{10}O_6$, is a tetrahydroxyflavone. The energetic action of alkali yielded phloroglucinol and protocatechuic acid, confirming Rochleder's results (*Zeit. fur Chem.*, 1866, ii., 602), and it is now found that, by gentler treatment, phloroglucinol and a substance having the composition $C_8H_8O_3$ are formed. (C = 63·16; H = 5·26. Found C = 63·47; H = 5·61). The substance (colourless needles, melting-point 115—116°) gives, with fused alkali, protocatechuic acid, and its properties are identical with those of *acetylcatechol*, $CH_3CO{\cdot}C_6H_3(OH)_2$ (Dzerzgowski, *Journ. Russ. Chem. Soc.*, 1893, xxv., 157). This confirms the constitution assigned by the author to luteolin,—



and is identical in its nature with the decompositions of chrysin, $C_{15}H_{10}O_4$ (Kostanecki, *Ber.*, 1893, xxvi., 2901), and apigenin (*Trans.*, 1897, lxxi., 805), which respectively yield phloroglucinol and acetophenone, and phloroglucinol and *p*-hydroxyacetophenone.

In conjunction with Mr. L. H. Horsfall, an investigation is in progress upon certain ethers of luteolin. By partial methylation, a *dimethyl ether* (found C = 65·06; H = 4·55; CH_3 = 9·92), colourless needles, m. p. 227—229° is formed in small quantity; this is almost devoid of dyeing property, and by means of alkali gives *isovanillic acid*. There is evidence that, in addition to luteolin, weld contains a trace of a second colouring matter.

171. "*The Action of Chloroform and Potassium Hydroxide on Orthoamidobenzoic Acid.*" By Walter J. Elliott, M.A.

By acting on orthoamidobenzoic acid with chloroform and potassium hydroxide, acidifying the product with acetic acid, and treatment with phenylhydrazine and semicarbazide respectively, the author has obtained the phenylhydrazone and semicarbazone of an aldehydo-orthoamidobenzoic acid. Owing to its great solubility in water, he has failed to obtain the free aldehydo-acid in a state of purity. The barium and silver salts of the phenylhydrazone have been prepared and analysed. The hydrazone and semicarbazone are both very stable.

172. "*Azo- and Hydrazone Compounds Differentiated by Bromine.*" By H. E. Armstrong.

Although the constitution of the "oxyazo"-compounds has been the subject of numerous investigations, apparently chemists are not yet in agreement as to their formulæ; indeed, only recently, Hantzsch in Germany, and MacPherson in America, have arrived at diametrically opposite conclusions. The difficulty arises from the fact that parahydroxyazobenzene—the parent of all "oxyazo"-compounds—and quinonephenylhydrazone are unquestionably isodynamic. Methods such as Hantzsch and Farmer have adopted must, therefore, be regarded as in principle the only suitable ones for the determination of structure in such cases, and extreme care must be taken in intrepreting interactions.

A number of observations made in the author's laboratory by Messrs Panisset, Seligmann, and Isherwood are of interest as bearing on the problem.

The stability of diazobenzene in presence of bromine was established by Griess in his earliest investigation. Diazobenzeneparasulphonate is also untouched by bromine, and action only takes place gradually as it becomes hydrolysed. Azobenzene is only very slowly and imperfectly acted on by bromine. Nitrogen, in fact, by no means tends to promote substitution, unless associated with hydrogen.

If oxyazobenzene were parahydroxyazobenzene, one would expect it to be readily brominated in the ortho-position relatively to the hydroxyl; but, as a matter of fact, it yields a product convertible into parabromaniline and phenol on reduction, and which is easily prepared from these, a result only compatible with the view that towards bromine it behaves as a hydrazone.

This view is confirmed by the fact that when the ethylated compound is brominated, a substance is formed which Mr. Isherwood finds to be identical with that produced on coupling orthobromophenol with diazobenzene and then ethylating. It is noteworthy that the bromine is removed from this compound with exceptional facility, both when dissolved in acetone and when left in contact with sulphurous acid.

The behaviour of bromine in excess towards the quinhydrazones is characteristic. Quinonebromophenylhydrazone is resolved by it into bromodiazobenzene and tribromophenol, and ordinary tropæolin into diazobenzenesulphonate and tribromophenol. So complete is the change in this latter case, that if, after the tribromophenol has been filtered off, phenol be added to the solution, and then alkali, an amount of tropæolin can be recovered almost equal to that originally taken.

Victor Meyer Memorial Lecture.

The Victor Meyer Memorial Lecture will be delivered by Professor T. E. Thorpe, President of the Society, on the evening of Thursday, February 8, 1900, at 8.30.

The Decomposition of Alkaline Hypochlorites.—J. Thomsen.—The author shows that some of the results recently obtained by Foerster and Jorre agree with those already obtained by him (*Thermochem. Untersuch.*, ii., 123). The aqueous solutions of hypochlorous acid, as well as solutions of hypochlorite of soda containing an excess of acid are rapidly decomposed.—*Journ. für Prakt. Chem.*, 1899, lix., p. 244.

CORRESPONDENCE.

RELATIONS BETWEEN THE ATOMIC WEIGHTS AND PHYSICAL PROPERTIES OF ELEMENTS.

To the Editor of the Chemical News.

Sir,—In my note (Chem. News, lxxx, p. 314) I pointed out that the product of melting-point (absolute) × coefficient of linear expansion for 1° C. is a constant for a number of elements, and that this product has higher and varying values for several other elements. The coefficients were (as I indicated) determined by various observers and at various (disproportionate) temperatures, but they were the best available. I did not attempt to explain the different values of the product.

The idea of absolute zero and the absolute scale of temperature arose from the study of the properties of the permanent gases (as they were then considered) at an early period in the history of modern science. But, as long ago as 1848, Wm. Thompson (now Lord Kelvin) based a scale of absolute temperature upon the fundamental principles of thermodynamics, and showed that the zero of this scale is really absolute,—that is, if it is zero for one substance it is zero for every other. Lord Kelvin and Joule have since determined experimentally, by means of the cooling effect produced in a fluid when forced through a small orifice or porous plug, that zero on this scale coincides with that originally deduced from the thermal expansion of gases. The results of their experiments on hydrogen, air, and CO_2, gave the absolute temperatures of melting ice as 273°, 273·14°, and 273·9° respectively.

This refers the expansion of gaseous bodies to absolute zero as deduced on independent mathematical grounds: and as showing that a physical property of solids is referable to the same starting-point, the experiments of Dewar and Fleming may be mentioned. These investigators found that the electrical resistance of certain pure metals, of which platinum is one, diminishes at low temperatures in such a way as to show that it tends to vanish at −273° C. At this temperature all heat has disappeared, and consequently the resistance caused by it.

It therefore appears as if the title which Mr. Wertheimer writes of as "only a survival from the infancy of modern chemistry" is likely to survive well into the twentieth century, whether this begins in 1900 or 1901. In asserting a different value to the absolute zero for zinc and cadmium he seems necessarily to be assigning a positive value to zero in one instance or the other.

The germ of a valuable idea is contained in Mr. Wertheimer's references to the coefficient of expansion taken at "similar" and "similarly located" temperatures. For just as it is necessary to measure the coefficient of a gas at a sufficient distance above its condensing point, so it is necessary to determine the coefficient of a solid at a point removed, and probably proportionately removed, from the melting-point.

As regards Mr. Wertheimer's suggestion that the coefficient of expansion × B.P. − M.P. might give universal constants, it must not be overlooked that boiling-points, unless taken in an absolute vacuum, must be arbitrary. Ordinarily they are given for the atmospheric pressure that happens to prevail on this planet. If interesting results should be found by this means, it is probable they would be due to the nature of *c*, where B.P. *in vacuo* × *c* = B.P. at 760 m.m.

The study of melting-points, boiling-points, and atomic volumes, by the graphical method, shows an interrelation to exist between these properties of the elements, —*e.g.*, B.P./M.P. Zn = B.P./M.P. Cd.—I am, &c.,

Thomas Bayley.

January 1, 1900.

NOTICES OF BOOKS.

Industrial Electricity. Translated and Adapted from the French of Henry de Graffigny, and Edited by A. G. Elliott, B.Sc. London: Whittaker and Co. Pp. 152. With 65 Illustrations.

The volume now before us is the first of a Series which it is proposed to issue on the subject of Electro-mechanics. This series is likely to be popular, inasmuch as the explanations are in clear and non-mathematical language. The present volume may be considered as introductory to the series, each chapter—of which there are XII.—dealing shortly with a separate branch of practical electricity.

The other volumes of the series will treat the more important branches of the subject separately and in detail.

A Synopsis of the British Pharmacopœia, 1898. Compiled by H. Wippell Gadd, with Analytical Notes and Suggested Standards by C. G. Moor, M.A., F.I.C. Fourth Edition. London: Baillière, Tindall, and Cox. 1899. 223 pp.

Now that the Pharmacopœia has been in use for over twelve months it has been thought desirable to omit the tables of alterations, additions, and omissions in this the fourth edition of the "Synopsis." The changes will be still found mentioned, however, in the general table, to which also specific gravities, alcoholic strengths, and percentage of solid residues have been added in many cases.

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NOTICE IS HEREBY GIVEN, that Oliver Imray, of Birkbeck Bank Chambers, Southampton Buildings, Chancery Lane, in the County of London, has applied for leave to amend the Specification of Letters Patent No. 21839 of 1898, for the "Manufacture of Dyestuffs of the Diphenylnaphthylmethane Series."

Particulars of the proposed Amendment were set forth in the "Illustrated Official Journal" (Patents) issued on the 30th December 1899.

Any person, or persons, may give notice of opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

(Signed),
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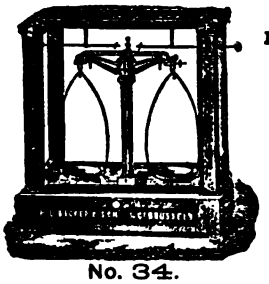

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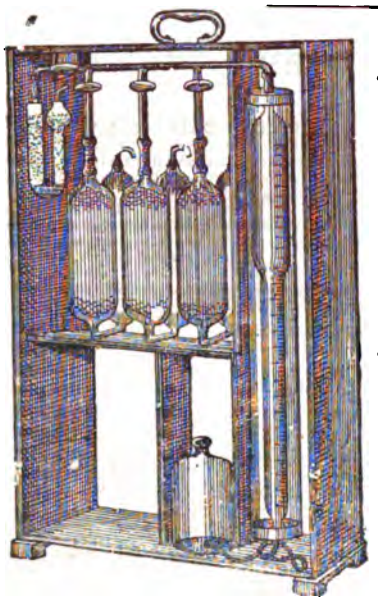

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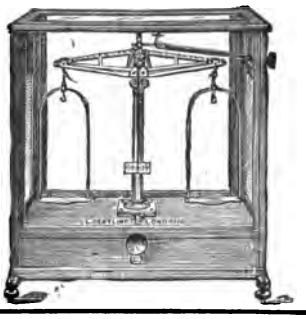

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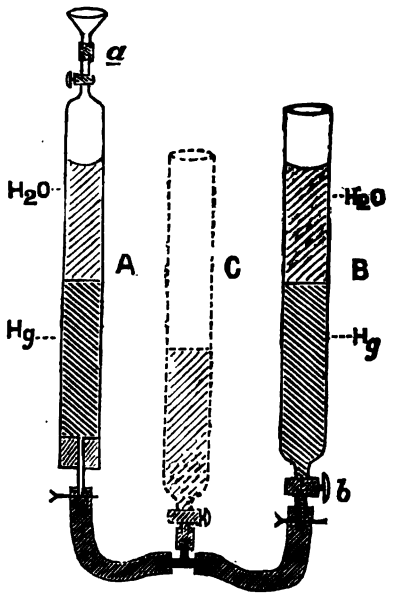
LABORATORY NOTES.

On the Solubility of Some Gases in Water.

By WILLIAM FRENCH, M.A., and F. ASHWORTH, B.A.

The apparatus in use for the determination of the solubility of the more sparingly soluble gases, oxygen, nitrogen, &c., viz., that of Bunsen, and Heidenhain and Meyer, whilst admirably adapted for working where great accuracy is required, seemed to one of us to be quite unnecessary for ordinary class work, or, when it is required to demonstrate (1) that these gases are appreciably soluble in water, (2) that oxygen is more soluble than nitrogen. The object of these experiments were taken, therefore, not with a view of confirming the results obtained by previous workers, but rather to see how far ordinary apparatus, such as will be found in any well-equipped laboratory, could be adopted for the purpose.



Our first experiments were carried out with a Lunge's nitrometer, and the numbers (representing solubilities) obtained approximated so near published numbers, that we were induced to repeat them with a piece of apparatus constructed from two (or three) burettes. The method of working was essentially the same whichever apparatus was used, with the exception that no funnel was required with a nitrometer.

A is an inverted stoppered burette, fitted with an india-rubber stopper, and connected by pressure tubing to another burette, B. A small funnel is fitted at A with rubber, and mercury is placed in B, which is raised until the metal begins to pass into the funnel; cold, well-boiled distilled water is added to the funnel, and B quickly lowered, so that about 30 c.c. of water pass into A; the funnel is removed, and the gas supply attached to A, and about 5 c.c. of the gas drawn in. Water is now added to B, so that the heights of the water in each side is the same *when the mercury levels are the same* (the reason of this is obvious), and the volume of water and gas taken quickly read off. The stopcock B is now closed (or the tubing is pinched) and the burette A is well shaken; B is then opened, the levels equalised, and the diminution of gas noted; this is repeated until the diminution is constant. The volume of gas dissolved is reduced for temperature and pressure, and the solubility obtained in the usual way. A burette C (in dotted lines), fitted with a T-piece into the pressure tube, greatly facilitates quick working, it forming a reservoir for the mercury, but it is not essential. Using this apparatus for class work, students working in pairs may quite readily prove the statement so often made in text-books that oxygen is more soluble in water than nitrogen, and using great care very fair results may be obtained. The following numbers were obtained by Messrs. Howlett, Evans, Macfarlane, and Garnett; beneath these are numbers taken from "Bayley's Chemist's Pocket Book" for the same temperatures for comparison:—

Oxygen.	Nitrogen.	Air.	CO_2 found.
0·02879 15°C.	0·0151 15°C.	0·01729 19°C.	0·952 15°C.
0·02989 ,,	0·01478 ,,	0·01719 ,,	1·002 ,,

Grammar School, Bury.

THE SEPARATION OF TUNGSTEN AND MOLYBDENUM.

By FRED IBBOTSON and HARRY BREARLEY.

Of the few methods one can find for effecting this separation, volatilisation of the molybdenum in a stream of hydrochloric acid gas is best known, and appears to be most commonly used. As a rule, tube volatilisations are made use of in metallurgical laboratories only as a last resource or for research purposes. On this account a separation which can be made in every-day apparatus and with common reagents was desirable.

Quite a number of circumstances can be arranged under which molybdenum and tungsten behave in diverse ways, but very few in which the behaviour, when the two elements are together, of the one is so sharply different from that of the other as to provide an accurate means of separating them. These methods chiefly depend on the solubility of the one and the insolubility of the other metal compounds (generally oxides) in certain acids, and they are deficient because the WO_3 is never perfectly insoluble, nor the MoO_3 perfectly soluble, except when alone.

The following experiments were made with the object of determining with what degree of accuracy one or two theoretically sound principles could be applied.

I. *Volatilisation of Molybdic Oxide in the Muffle.*

MoO_3 can be completely volatilised in an ordinary gas muffle if alone, and very approximately, though with difficulty, when precipitated along with WO_3.

Sodium tungstate, equivalent to 0·1340 grm. WO_3, and sodium molybdate, equivalent to 0·1500 grm. MoO_3, were precipitated with mercurous nitrate and mercuric oxide and ignited in the muffle. Long after the mercury had been vapourised white fumes of MoO_3 came from the crucible, but ceased to be visible some time before nearly all the molybdenum was got rid of. Ninety per cent of the Mo can be easily volatilised; the remaining 10 per cent needed several hours at a full red heat to drive it off. After reaching constant weight 0·1360 grm. WO_3 remained. According to Read (*Journ. Chem. Soc.*, lxv., 313), WO_3 is stable at 1750° C. It might be easy if such temperatures were available to expel the MoO_3, but even then it would be troublesome to adapt the process so as to estimate the molybdenum directly.

II. *Separation with Hydrochloric Acid Solution.*

Precipitated amorphous MoO_3 is easily soluble in warm hydrochloric acid, but the variety, glistening and wool-

like, obtained by sublimation is not soluble in hydrochloric acid nor in *aqua regia*. This may account for the failure of attempts to separate ignited WO_3 and MoO_3 by treatment with HCl.

The solution of an alkaline molybdate, unless very strong, is not precipitated by adding hydrochloric acid; an emulsion of lead molybdate in water is completely dissolved by nitric acid; ignited lead molybdate is dissolved by hydrochloric acid, and not precipitated at all on diluting. On the other hand, WO_3, after baking or igniting, is insoluble in HCl; the solution of an alkaline tungstate, if not too dilute, is precipitated by HCl; an emulsion of lead tungstate is decomposed by nitric acid with precipitation of WO_3; ignited lead tungstate is completely precipitated as WO_3 by diluting its solution in strong HCl, with two or three volumes of hot water.

Some of the above statements respecting tungsten are not absolutely true; they are so far from being true in fact, that in delicate analytical operations account must be taken of the errors which would creep in were they taken to be so. For instance, WO_3 is never absolutely insoluble in HCl. Prepared by decomposing an alkaline tungstate, or by diluting a hydrochloric acid solution of lead tungstate with water, WO_3 may be re-dissolved to a clear solution by concentrated HCl. After evaporation to dryness and baking, the solubility is less marked but still very appreciable. These facts are less obvious to casual observation, because nearly all the WO_3 is precipitated again on dilution. Even ignited WO_3 is not quite insoluble. By treating 0·4980 grm. ignited WO_3 with 60 c.c. strong HCl three times successively there were found:—

In Residue.	In Filtrate.
0·4945	0·0030
0·4910	0·0027
0·4876	0·0030
Lost from Residue.	**Found in Filtrate.**
0·0104	0·0087

From mixtures of one or two decigrms. of molybdenum and tungsten in the form of alkaline salts, the WO_3 precipitated by an acid is usually from 5 to 10 per cent too high, a result due, of course, to contamination with MoO_3. The error is about the same if the moist $PbWO_4$ and $PbMoO_4$ are dissolved in HCl, and the WO_3 precipitated by diluting.

By re-dissolving the WO_3 in HCl and re-precipitating, the WO_3 generally weighs two or three m.grms. too little, due to the fact that at each precipitation a little WO_3 remains in solution, and that after the second precipitation less MoO_3 accompanies it than compensates for such loss. These small errors can never be entirely eliminated in any separation in HCl solution, but they may be minimised in either of the following ways:—

(A). Precipitate the Mo and W together by means of lead acetate, wash slightly with hot very dilute acetic acid, and ignite. Transfer to a beaker and dissolve in concentrated HCl, using about as many c.c. of acid as centigrms. of the lead salts.

The precipitates are most conveniently dissolved by heating just short of the boiling-point of the acid; in this way very few irritating fumes are evolved, and the acid is not weakened to any considerable extent before complete solution has been effected. To the clear or possibly faintly opalescent solution, add two or three times its bulk of boiling water, boil, allow to settle, decant clear liquid through a small pulp filter, re-dissolve in about half the quantity of acid previously used, re-precipitate with water, and filter through the same pulp. The Mo is determined in the filtrate, either with or without removing the HCl, by precipitation as $PbMoO_4$ (Chem. News, lxxviii., 203).

A number of experiments, which need not be quoted, show that the complete decomposition of $PbWO_4$ can be relied on only by dissolving in strong HCl and precipitating by dilution; diluted HCl does not completely decompose $PbWO_4$ after ignition.

If twice as many c.c. of acid are used as there are centigrms. of the lead salts, the tungsten is completely precipitated by diluting to three times the volume of the solution. Generally, larger amounts of acid used to dissolve the ignited precipitates require proportionally larger amounts of water for complete precipitation of the WO_3. The estimation is not then so satisfactory, as the greater the dilution the greater the liability to contamination with MoO_3.

The process just described gave the following results:—

	Added.	Found.	Error.
Mo	0·0918	0·0908	−0·0010
W	0·1031	0·1037	+0·0006
Mo	0·0551	0·0549	−0·0002
W	0·1031	0·1028	−0·0003
Mo	0·0367	0·0374	+0·0007
W	0·1031	0·1023	−0·0008
Mo	0·0184	0·0193	+0·0009
W	0·1031	0·1017	−0·0014
Mo	0·0092	0·0092	0·0000
W	0·1031	0·1023	−0·0008

Where the amount of Mo is very small, as in the last sample, it is not necessary to precipitate the WO_3 twice,

(B). If the amount of W is small compared with the Mo, it is not by any means totally precipitated on diluting to the usual volume. The following method, which gives as good results under any circumstances as the preceding, should then be adopted:—

The HCl solution of the ignited lead salts, to which a few drops of HNO_3 have been added, is evaporated, preferably over the water-bath, until nearly pasty, when the WO_3 will have almost entirely separated. The mass is then diluted with 100 to 200 c.c. dilute HCl (3 water to 1 acid), the solution boiled, and the WO_3 filtered off. The weak acid precipitates any WO_3 still unseparated by the evaporation, and is yet strong enough to hold the molybdenum in solution.

Whether the evaporation be performed slowly over the water-bath, or more rapidly, some of the WO_3 always adheres to the vessel and cannot be cleared off. A good plan for estimating this consists in dissolving it in 2 or 3 drops of dilute ammonia. The solution is then absorbed by a piece of ashless paper which is ignited along with the main portion of the precipitate. Appended are some test analyses:—

Added.	Found.	
0·1310	0·1290	WO_3
0·3510	0·3500	$PbMoO_4$
0·0780	0·0760	WO_3
0·3510	0·3510	$PbMoO_4$
0·0520	0·0510	WO_3
0·3510	0·3515	$PbMoO_4$
0·0131	0·0120	WO_3
0·3510	0·3495	$PbMoO_4$
0·0050	0·0040	WO_3
0·3510	0·3500	$PbMoO_4$

In some notes on the estimation of tungsten by one of us (Chem. News, lxxix., p. 64), it is shown that a precipitate corresponding exactly to the formula $PbWO_4$ is not obtainable by adding lead acetate to a soluble tungstate when there is any excess of acetic acid present, but is invariably acid in character, nor is tungsten completely precipitated in the presence of excesses of acetates and chlorides. As these very conditions of precipitation are favourable to the complete precipitation of molybdenum as $PbMoO_4$, it was thought advisable, as a fitting conclusion to this paper, to see whether the precipitation of the two metals together under these circumstances would yield a mixture of *true* lead salts or not.

The following is a typical result:—

With an excess of 10 c.c. acetic acid, the weight of mixed lead salts was 0·5030, containing on analysis 0·0800 WO_3 instead of 0·0786, and 0·3500 $PbMoO_4$

instead of 0·3510 added. 0·0786 WO_3 represents 0·1541 $PbWO_4$, which, added to 0·3510 $PbMoO_4$, gives 0·5051 as the weight of mixed salts instead of 0·5030 actually got.

In the following analyses an amount of W corresponding to 0·1256 WO_3, and of Mo corresponding to 0·4256 $PbMoO_4$, were present in each case, so that, on the supposition of their being *true* lead salts, the weight of the mixed precipitates should be 0·6719 grm. For 0·1256 WO_3 represents 0·2463 $PbWO_4$, and 0·2463 + 0·4256 = 0·6719.

	Wt. mixed lead salts.	WO_3.	$PbMoO_4$.
15 c.c. acetic acid	0·6771	0·1248	0·4247
Sodium acetate corresponding to 10 c.c. acetic acid.	0·6690	0·1232	0·4232
Ammonium acetate corresponding to ditto	0·6760	0·1241	0·4221
Ammonium chloride corresponding to 10 c.c. strong HCl	0·6792	0·1278*	0·4252

Note.—The noteworthy feature of these results is the indication that in the presence of not less than an equal amount of MO, the W is not precipitated as an acid salt even when the solution contains considerable amounts of free acetic acid; and that the alkaline acetates and chlorides exert a comparatively insignificant solvent action under the same conditions. The feasibility of directly determining only one acid and calculating the other from the combined weight—$PbWO_4 + PbMoO_4$—has been questioned, but our results now show that the plan merits greater reliance than could be claimed for it in view of the difficulty of obtaining pure $PbWO_4$ when W is precipitated separately. They show, in fact, that the combined lead salts are actually somewhat basic and not acid.

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THE

ELECTROLYTIC ESTIMATION OF MANGANESE IN MANGANESE ORE.

By ARTHUR HIORNS, Chemist to the Usina Wigg Manganese Mines, Estacao de Miguel Burnier.

There are several solutions from which manganese may be estimated electrolytically, but for the estimation of the metal in the oxide, the acid solution is the most simple, accurate, and rapid. No matter which solution is used, the quantity of manganese in each solution to be electrolysed must be very small, and to obtain accurate results the quantity should never exceed 0·03 grm. of metal.

In the Usina Wigg ore, which has an average of about 55 per cent of metallic manganese, 0·05 grm. of the ore is ample for its proper estimation.

The acid solution is electrolysed hot, and the manganese dioxide which is deposited at the positive pole is converted by ignition to Mn_3O_4, which gives results varying only in the fourth decimal place.

The solution made strongly acid with nitric acid is contained in a previously weighed platinum dish, and a spiral of platinum wire is brought into contact with the surface of the liquid. The dish and spiral are connected to a Bunsen cell, the dish to the positive pole and the spiral to the negative pole.

The dish is placed on a water-bath, and as soon as the current is started heat is applied to the water-bath and the temperature kept as near 60° C. as possible. It is necessary to have the solution very acid to start with, and as during the operation it is turned into ammonia, a few drops of acid must occasionally be added to it. Great care is necessary to watch that the solution is always acid.

The whole of the manganese is deposited in twelve hours—ten hours seem to be sufficient—but passing the current for twelve hours makes the total deposition certain. The best mode of procedure after the current is stopped is to wash the MnO_2 very carefully with water, then dry it and ignite to convert the MnO_2 into Mn_3O_4.

The following are the results of the estimation of manganese in two samples of ore by this method. 0·05 grm. of ore taken in each case.

	Weight of Mn_3O_4. Grm.	Percentage of metallic manganese.
A.		
Electrolysed ten hours.. ..	0·0401	57·7800
Electrolysed twelve hours ..	0·0398	57·3500
Electrolysed fourteen hours..	0·0399	57·4959
B.		
Electrolysed ten hours.. ..	0·0374	53·8934
Electrolysed twelve hours ..	0·0376	54·1816
Electrolysed fourteen hours..	0·0373	53·7493

Average of A .. .. .. 57·5419 per cent
Average of B .. .. 53·9414 per cent

The volumetric method of estimation gave in these two cases—

A, 57·5512 per cent. B, 54·0036 per cent.

A CONTRIBUTION TO THE STUDY OF LIQUID MIXTURES OF CONSTANT BOILING-POINT.*

By GARNETT RYLAND.

Introduction.

In the course of an investigation undertaken for another purpose, a mixture of methyl alcohol and benzene was subjected to a fractional distillation, when it was observed that the larger part boiled like a homogeneous liquid at 57—58°, and could not be separated by repeated distillation with a Hempel tube. By making trial mixtures it was found that, mixed in certain definite proportions, methyl alcohol and benzene will boil to the last drop at 57—57·5°, under atmospheric pressure. If there is present an excess of either of the constituents, the constant boiling mixture and this excess are separated by fractional distillation. As roughly determined, the proper ratio seemed approximately $3CH_4O : 2C_6H_6$ (38·1 parts of alcohol to 61·9 parts of benzene, by weight).

On examination of the literature no such observation of this mixture could be found, though several similar phenomena are on record.

Historical Sketch.

1. H. E. Roscoe ("On the Composition of Aqueous Acids of Constant Boiling-point," *Journ. Chem. Soc.*, 1861, xiii., 146, and 1862, xv., 270) investigated certain aqueous acids of constant boiling-point which had been regarded as definite hydrates, and found that the proportions of water and acid were not those required by any simple molecular ratio, nor did the composition remain the same when the pressure varied.

Aqueous nitric acid, when distilled under a pressure of 735 m.m., always gave a residue which boiled at 120·5°, and contained 68 per cent of pure acid. Distilled under 75 m.m. pressure, the per cent of pure acid was 66·7, and under 1260 m.m. it became 68·8.

Aqueous solutions of hydrochloric, hydrobromic, and hydriodic acid gave similar phenomena, only in these cases the per cent of acid increased as temperature and pressure decreased. Hydrofluoric acid resembled nitric acid in all respects.

Aqueous formic acid containing 77·5 per cent of pure acid (boiling-point 101·1°) boiled at the fixed temperature

* The amount of WO_3 recovered when the W was precipitated alone was only 0·0980 grm. instead of 0·1256 grm. The great influence exerted by the co-precipitated molybdenum is seen on comparing these results with those previously given (Chemical News, lxxix, p. 64).

* From the Dissertation submitted by the author to the Board of University Studies of the Johns Hopkins University for the degree of Doctor of Philosophy, June, 1898. The investigation was undertaken at the suggestion of Professor Remsen, and carried on under his supervision. From the *American Chemical Journal*, vol. xxii., No. 5, Nov., 1899.

of 107·1°, under 760 m.m. pressure, the proportion of acid increasing with the pressure.

Aqueous perchloric acid ("On Perchloric Acid and its Hydrates," *Proc. Roy. Soc.*, 1862, xi., 493) gave a residue which boiled constantly at 203°, under atmospheric pressure, and contained 71·6 per cent pure acid.

2. Alexander Bauer ("Ueber eine merkwürdige Erscheinung bei der Destillation eines Gemenges von Bromäthylen und Brompropylen," *Ann. Supp.-Band.*, 1861, i., p. 250) found that a mixture of ethylene bromide (boiling-point 129°) and propylene bromide (boiling-point 141°) in equivalent amounts, distilled at 134°), and could not be separated by fractionation.

3. J. A. Wanklyn ("Ueber die Destillation von Gemischen; ein Beitrag zur Theorie der fractionerten Destillation," *Liebig's Ann. Chem.*, 1863, xxviii., p. 328) called attention to the fact that in the distillation of a mixture of liquids the proportions in which the constituents pass over depend not merely on their relative quantity and on their respective vapour-tensions at the temperature of ebullition, but also upon their mutual adhesion and on the density of their vapours.

On distilling a mixture of 18 grms. of methyl alcohol (boiling-point 66°) and 17 grms. of methyl iodide (boiling-point 72°), the first third of the distillate contained a greater amount of the less volatile than of the more volatile liquid.

4. Berthelot ("Sur la Distillation des Liquides melangés," *Ann. Chim. Phys.*, 1864 [4], i., 384) stated that a mixture of 91 parts carbon disulphide (boiling-point 48°) and 9 parts alcohol (boiling-point 78°), by weight, boils at 43—44°, remaining at this temperature from the beginning until the end of the distillation. A mixture of this composition is separated by fractional distillation from an excess of either of its components.

He found the explanation of this phenomenon in essentially physical ideas. If a mixture of two liquids which do not exert any reciprocal action is boiled, both will be volatilised at that temperature at which their united vapour-tensions equal the atmospheric pressure, and in the same ratio by weight as that of the products of their actual vapour-tensions by their vapour-densities respectively. So if the carbon disulphide and alcohol preserved their theoretical vapour-tensions, they would be distilled at about 40° in the proportions of 88·5 parts of carbon disulphide and 11·5 parts of alcohol. But the two liquids exert influence upon each other, as is shown by their mutual solution and by the diminution of the sum of the tensions of their vapours. This influence tends to diminish the individual vapour-tension of each of the liquids, following an unknown law.

5. Ed. Linnemann ("Untersuchung über die Beziehungen des Isopropyl Alcohols zum Propyl Glycol und zum Glycerin," *Liebig's Ann. Chem.*, 1865, cxxxvi., p. 40) records three hydrates of isopropyl alcohol (boiling-point 83—84°, 739 m.m.): $3C_3H_8O+2H_2O$, formed when aqueous propyl alcohol is distilled on the water-bath, boils at 78—80°, 738 m.m., specific gravity 0·832, at 15°; $2C_3H_8O+H_2O$, formed by dehydration with potassium carbonate, boils at 80°; $3C_3H_8O+H_2O$, formed by distilling from anhydrous copper sulphate, boils at 81°, specific gravity 0·800.

6. T. E. Thorp ("A Contribution to the Theory of Fractional Distillation," *Journ. Chem. Soc.*, 1879, xxxv., p. 544) found that a mixture of 1 part methyl alcohol and 3·6 parts carbon tetrachloride boils like a homogeneous liquid about 10° lower than methyl alcohol, the more volatile of the two liquids. Calculation shows that this ratio is almost identical with that obtained by multiplying the vapour-tensions of the two liquids at the temperature of the boiling-point (55·7°) by their respective vapour-densities.

7. D. Konowalow ("Ueber die Dampfspannungen der Flüssigkeitsgemische," *Wied. Ann.*, 1881, xiv., p. 34) investigated the known mixture of propyl alcohol (boiling-point 97·4°) and water in the ratio of about 77 : 23 parts, which boils at 85—85·5°. To this composition corresponded the maximum of the curve, whose abscissæ are percentage quantities and ordinates pressures, representing the vapour-tension of the mixture at a specific temperature.

He found also that a 25 per cent. solution of butyric acid (boiling-point 163°) in water boiled constantly at 99·5°. Both of these mixtures change their respective proportions on change of the pressure and temperature of distillation.

8. Ramsay and Young ("Note on the Mixture of Propyl Alcohol and Water," *Abs. Proc. Chem. Soc.*, 1888-1889, p. 101) agreed with Konowalow that the mixture of propyl alcohol and water, thought by Chancel to be $C_3H_8O+H_2O$, which distils to the last drop at 87·5° (738 m.m.), is not a definite hydrate. The normal composition of the mixture (71·46 per cent alcohol to 28·54 per cent water) varied with the pressure under which it was distilled.

Apparatus for Distillation under Reduced Pressure.

Mixtures of methyl alcohol and benzene were distilled under different pressures, and the relative amounts of the two liquids contained in the constant boiling portion of the distillates were determined.

The apparatus used to effect fractional distillation under reduced pressure was arranged as follows*:—

A 250 c.c. separatory funnel was used as a receiver, This was closed with a two-hole rubber stopper, through which passed the adapter and a branched tube, the latter connecting with the manometer, the exhaust, and the pressure regulator. The last was a glass tube about 800 m.m. in length and 20 m.m. in diameter, filled to the necessary height with mercury, and closed at the top with a two-hole rubber stopper through which passed a short and a long piece of small glass tubing, The first connected with the receiver and the latter extended below the surface of the mercury a distance corresponding to the desired diminution of pressure. By this means the pressure could easily be kept constant within 10 m.m without interrupting the course of distillation. Successive fractions were drawn off from the funnel into an exhausted receiver, through whose stopper the stem of the funnel passed.

The liquid was distilled from a bulb provided with an additional side tube, connected by rubber and a pinch-cock with a small funnel, into which successive fractions were poured. With caution these could be introduced without diminishing the exhaustion.

(To be continued).

NOTES ON

THE ELECTRO-DEPOSITION OF CHROMIUM.†

By SHERARD COWPER-COLES, M.Inst.M.M.

Chromium does not occur in the free state, being generally found as an oxide; its lower oxides closely resembling those of iron, CrO and Cr_2O_3. Occasionally the metal occurs in a higher state of oxidation, in combination with lead, as plumbic chromate, $PbCrO_4$. It was in this mineral that Vauquelin, in the year 1797, first discovered the existence of chromium.

Chromium forms the colouring matter of several compounds, for instance, the green colour of the emerald, as was shown by Vauquelin; serpentine also owes its colour to chromium. Some of the characteristics of fused chromium are its great hardness (electro-deposited chromium is found to be comparatively soft), its high melting-point, it being more difficult to fuse than platinum, the temperature required being 2000° F.

* My thanks are due to Dr. E. E. Reid, of this laboratory, for his kind assistance in designing and constructing this apparatus.

† Read before the Institution of Mining and Metallurgy, Wednesday, December, 20th 1899.

The following table gives the comparative hardness of electro-deposited chromium, as represented by the number of centigrms. weight on a diamond point, required to produce a scratch:—

	Hardness.
Nickel electro-plate	10·0
Sheffield plate	10·0
Antimony electro-plate	9·0
Palladium (deposited bright)	9·0
Chromium deposited on copper	7·0
Palladium electro-plate	6·6
Cadmium-silver alloy, 5Ag, 39·5	5·0
Cadmium (deposited bright)	4·5
Silver (burnished)	4·0
Silver (chemically precipitated)	2·0

Chromium is little affected by nitric acid, even when hot and concentrated, pure chromium oxidising very slowly in hot air, but on heating the surface it becomes readily covered with a thin film of oxide; cold dilute sulphuric acid only attacks it slowly (Roscoe and Schorlemmer).

Some experiments made to determine the relative tarnishing of chromium as compared to other metals gave the following results. The amount of light reflected was determined by means of a Bunsen photometer and two pentane lamps. Highly polished reflectors were coated with various metals, and tested in the photometer, and then placed about 1½ inches from a small arc light for 4½ hours, when readings were again taken, with the following results:—

	Before tarnishing. Relative reflecting power.		After tarnishing. Relative reflecting power.	
	Unit law.	Square law.	Unit law.	Square law.
Silver (standard)	100	100	—	—
Chromium	100	100	56	27
Palladium	72	48	47	19
Platinum	56	26	44	16
Silver	111	105	95	90

Relative Diminution of Reflecting Power, due to Tarnishing.

	Unit law.
Chromium	44
Palladium	35
Platinum	22
Silver	10

Professor Hittorf found that chromium in dilute acid solutions in the cold causes hydrogen to be given off, but that chromium is electro-negative to such metals as copper, mercury, and silver. In the same way it fails to reduce such metals from solutions of their neutral salts, and behaves generally as an inert noble metal. On making chromium an anode in cold dilute acids, it dissolves, but not with the formation of chromic or chromous salts: it passes at once into the state of chromic acid. The results of the experiments carried out by Professor Hittorf are thus summarised by him:—Chromium can dissolve electrolytically in any one of its three states, CrO, Cr_2O_3, or CrO_3, according to the nature of the electrolyte and its temperature, its inactive state corresponding, perhaps, with the passive state of iron. The attainment of this passive state is not to be attributed to the presence on the metal of a thin layer of oxide, the hypothesis which is plausible for iron. It appears rather to imply that the surface, at least, of the metal differs in molecular arrangement according to the conditions and surroundings to which it is exposed: in short, that chromium appears to be an allotropic element. Chromium exists in three different states, viz,, passive, active, and intermediate, and in each of these states it occupies a different position in the electro-chemical series, and possesses a different chemical behaviour.

Metallic chromium has been obtained by the reduction of oxides and chlorides. Deville made a mixture of chromic oxides and sugar, which he intensely heated in a lime crucible, or heated chromium sesqui-chloride, with metallic zinc, under a layer of sodium chloride, the metal separating out in glistening scales.

A method of preparing chromium in the electric furnace has been devised by Herr Eschermann, in which a gas-tight steel furnace is used in connection with a movable electrode. A graphite crucible is placed in the furnace, and in this are placed oxide of chromium and sulphide of antimony, in the proportion of 10 to 23. After the furnace is closed, a comparatively small current of from 20 to 23 ampères is passed through the material until melted. Reaction takes place with the development of heat. The resulting mass consists of a regulus of chromium and antimony alloys, whilst the upper part of the crucible has an amorphous mass of antimony oxide, sulphide, &c., adhering to the walls. Every trace of antimony can be removed by repeated meltings, or by powdering and roasting at a white heat. The chromium dissolves a considerable portion of carbon, which separates out as graphite during cooling.

Metallic chromium has lately been obtained in considerable quantities by Dr. Hans Goldsmidt by reducing chromium oxide (Cr_2O_3) with aluminium in a fine state of division, the heat of combination being used for reducing the chromium oxide, the action being started by a small fuse or cartridge composed of peroxide of barium or other suitable per salt, which is fired by a piece of magnesium ribbon to start the reaction. Pure chromium is being made by an electrolytic process by the Electro-chemischen Werke, Bitterfield, Saxony. Chromium is being sold in Germany at the following prices:—Free from carbon, but containing some iron and traces of silica, 15s. a pound. Chromium free from carbon, and containing 96 per cent chromium, and 3 to 4 per cent iron and traces of silica, at 4s. a pound. Fused chromium is quoted on the American metal market at 7·14 dollars per kilogrm., and commercial pure powder at 1·90 dollars per kilogrm. Some interesting figures as to the production and value of chrome ore are to be found in the *Mineral Industry* for 1899.

Chromium carbide has been produced by Henri Moissan, the composition being Cr_3C_2 or Cr_4C. Chromium carbide is not acted upon by water; it forms an opaque crystalline mass with a metallic lustre.

Some attention seems to have been recently given to the electro-deposition of chromium from aqueous solutions. The process of Placet Barnet has been worked on a commercial scale. According to the patent specification, the electrolyte is made up by dissolving 100 parts of chromic alum in 100 parts of water by weight, 10 to 15 parts of barium bisulphate. The writer has not obtained deposits from this electrolyte when worked at various temperatues, using carbon and platinum anodes, with current densities varying from 15 to 19 ampères per sq. ft., the voltage at the terminals of the cell being about 4.

Bunsen experimented with the deposition of chromium, and succeeded in getting a good deposit of metal, which had the appearance of iron, but was less alterable in moist air, and even resisted the action of boiling nitric acid, but was acted upon by dilute sulphuric acid and hydrochloric acid.

E. Placet claims to have obtained beautiful deposits of chromium by electrolysing an aqueous solution of chromic alum, to which he added an alkaline sulphate and a small quantity of sulphuric acid.

The electro-refining of chromium has been tried by the firm of Messrs. Fred. Krupp, Essen, the object being to produce chromium free from carbon, no precautions being taken to prevent the production of a metal containing iron or ferro-chromium. Double chlorides of the metals are used as an electrolyte, the anodes being of ordinary carbonised ferro-chromium, porous diaphragms being used to separate the anode and cathode compartments.

Attempts have been made to deposit chromium alloys,, and a patent was obtained in March, 1884. Alloys of chromium were prepared by heating chromium compounds

with charcoal in a closed crucible, and pouring upon the reduced mass 2½ parts of copper, and, subsequently, from 1 to 1½ parts of molten tin, and then granulating, re-fusing, and casting in moulds of the desired forms. The plates thus formed were used as anodes in a solution made by dissolving 1 lb. of cyanide of potassium and 1 lb. of carbonate of ammonia in 1 gallon of water heated to 150° F., until a good deposit was obtained upon the cathode, a low current density being employed.

When a solution of chromium chloride is electrolysed with a negative electrode of mercury, the mercury is found to take up but little chromium, a brownish-black chromium oxide being formed in the liquor. T. Feree has used with mercury a solution composed of 160 grms. of crystallised chromium chloride, with 100 grms. of concentrated hydrochloric acid, 740 grms. of water, with a current of 22 ampères. In this way he found it possible to obtain 1½ kilos. of solid chromium amalgam in a very short time.

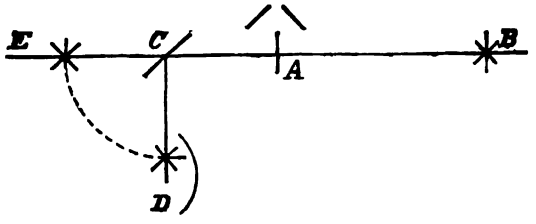
The writer has succeeded in obtaining beautiful bright deposits from a solution composed of chromic chloride 25 parts, water 75. The solution was worked hot at a temperature of 190° F., with a current of between 40 and 50 ampères per square foot. In the cold no deposit of metal was obtained, but a black non-adherent deposit, the voltage at the terminals of the cell being about 4. Gas was evolved at both anode and cathode during the deposition of the metal. Before good deposits could be obtained it was found necessary to add an excess of hydrochloric acid to dissolve the precipitated oxide and form a clear solution.

No deposit of metal could be obtained from a solution composed of chrome alum 100 parts, water 100 parts, barium sulphate 12 parts; the solution was worked hot and cold with current densities varying from 15 to 90 ampères per sq. ft.; a carbon anode and copper cathode were used.

On the table are specimens of electro-deposited chromium; some of the specimens have not been polished, but represent the deposited metal as thrown down on a silvered-copper surface.

The following table gives the actual and comparative amount of light reflected from various electro-deposited metals, including chromium; a Bunsen photometer and standard pentane lamps were used:—

Metals.	Distance from standard light to screen. Direct light (*a*).	Reflected light (*b*).	Reflecting power. $\left(\frac{a}{b}\right)\cdot\left(\frac{a}{b}\right)^2$.	
	Actual.			
	C.m.	C.m.		
Silver	69·5	71·5	Actual reflecting powers	0·97 0·94
Chromium ..	69·5	71·5		0·97 0·94
Platinum ..	69·5	97		0·72 0·52
Palladium ..	69·5	112		0·62 0·38
	Comparative.			
Silver	100	Reflecting power.		
Chromium ..	100	,,	,, $\left(\frac{a}{b}\right)$, taking Ag as 100.	
Platinum ..	74	,,	,, ,,	,,
Palladium ..	65	,,	,, ,,	,,



To measure the reflecting power of the various metals, the two standards of light were first compared (E and B) and the measurements taken. The reflecting surface was then placed in position at C and the lamp at E moved round to D, keeping the length of path of light constant; the lamp B was adjusted and a measurement again made.

ON THE

SEPARATION OF FERROCYANIDES FROM CARBONYLFERROCYANIDES, AND THE ESTIMATION OF THESE COMPOUNDS.

By J. A. MULLER.

I. *Estimation.*

a. Ferrocyanides.—To a solution of ferrocyanide (4 or 5 grms. per litre), slightly acidulated with nitric or acetic acid, nitrate or acetate of lead is added until a precipitate is no longer formed. After twelve hours the precipitate is filtered and washed as thoroughly as possible with boiling water; it is then dried and detached from the filter and treated with an excess of a mixture of 5 volumes of concentrated sulphuric acid and 3 volumes of water. The crucible is covered and heated, at first gently, on a sand-bath, then more strongly until the excess of sulphuric acid is driven off. The crucible is then placed in a muffle, and gradually heated to dull (not bright), redness; it is kept for some time at this temperature, then allowed to cool, and the residue, formed of a mixture of sulphate of lead and ferric oxide, is weighed. The filter, still impregnated with the precipitate, is treated in the same manner, and the weight of this residue is added to the first weight. In this second treatment care must be taken to moisten the residue with sulphuric acid after the preliminary heating, it is then calcined afresh.

The weight of the mixture of sulphate of lead and ferric oxide multiplied by 0·5378 gives that of the corresponding ferrocyanide of potassium.

By proceeding in this manner, I found in nitric solution 99·72 per cent, and in acetic solution 100·20 per cent.

b. Carbonylferrocyanides.—To the dilute solution acidulated with acetic acid, an excess of pure acetate of copper is added after twenty-four hours, filter,* and wash thoroughly; this operation takes a very long time. The precipitate, with the filter, is then placed in a large platinum crucible, and moistened with a mixture of sulphuric acid and water of the strength previously given; the crucible is covered and heated, at first very gently on a sand-bath, then more strongly to redness, so as to completely burn the carbon. The residue is dissolved in fuming hydrochloric acid, and from the previously diluted solution the copper is precipitated in the form of cupric sulphide, which is transformed, after desiccation, into cuprous sulphide by Rose's method.

The weight of Cu_2S, multiplied by 2·787, gives the weight of the corresponding carbonylferrocyanide of potassium.

By experiment I found 99·12 per cent.

II. *Separation.*

In a solution of pure carbonylferrocyanide of potassium, slightly acidulated with acetic acid, acetate of lead forms no precipitate, while the ferrocyanide is completely precipitated, as has been shown above in the state of ferrocyanide of lead.

But if the solution of the prussiate contains carbonylferrocyanide as well, the precipitate of ferrocyanide of lead formed contains a notable quantity of carbonylferrocyanide of lead.

The following is the way in which, after a large number of experiments, I succeeded in totally separating the two ferrocyanides:—

* The liquid does not pass clear until the filter is impregnated with the precipitate; the filtration is therefore excessively slow.

To the weak solution of the ferrocyanides (4 to 5 grms. per litre), slightly acidulated with acetic acid (8 to 10 grms. per litre), is gradually added, while stirring, a slight excess of a solution of acetate of lead; the mixture is then left in the dark until the supernatant liquid becomes clear; this takes from one to two days. The clear liquid is then decanted carefully with the aid of a pipette, and filtered several times until it is quite limpid; an excess of acetate of copper is then added for the purpose of precipitating the carbonylhydroferrocyanic acid in the state of a cupric salt. As for the plumbic precipitate, it is dissolved in a slight excess of a solution of pure soda, and the mixture is poured, while still stirring, into water containing enough acetic acid for the whole to be still acid after the addition of the liquid. After having washed the beaker containing the sodic solution with acidulated water, and adding the washings to the principal solution, the plumbic precipitate re-forms; when this latter is completely deposited it is left in the dark for twenty-four hours.

The above series of manipulations is then repeated, once not being sufficient.

After having made three successive solutions and precipitations of the plumbic precipitate, the latter is thoroughly washed and estimated as was described in *a*. As for the liquids separated after each precipitation and addition of acetate of copper, they are filtered, and the precipitate of carbonylferrocyanide of copper, after being well washed, is treated as in *b*.

By operating exactly as has just been described a mixture composed of—

$K_4Fe\overline{C}N_6$ .. .. 0·7348 grm.
$K_3FeCO\overline{C}N_5$ .. .. 0·6244 „

gave me—

$K_4Fe\overline{C}N_6$.. .. .. 0·7302 „
$K_3FeCO\overline{C}N_5$ .. .. 0·6267 „

that is 99·37 per cent of the first salt, and 100·37 per cent of the second.—*Bull. Soc. Chim.*, Series 3, vol. xxi., No. 10.

THE PREPARATION OF PURE ALKALINE NITRITES.

By E. DIVERS.

Nitric acid is made to react on starch, or on arsenious acid; the temperature and the strength of the acid are regulated in such a manner that in the products obtained nitric oxide should be in excess over the peroxide. The gas is passed into an empty flask, where the nitric acid is deposited, then into a concentrated solution of a pure alkaline hydrate or carbonate.

The nitrites of potassium and sodium are slightly yellow, very soluble in water, and give solutions alkaline to litmus. When free from nitrates their solutions may be evaporated to dryness without decomposition. The two salts when crystallised are anhydrous, but they soon deliquesce. Nitrite of sodium melts at 271°.—*Journ. Coll. Sci., Tokio, Japan,*

THE FORMATION OF HYPOCHLORITES AND CHLORATES.

By F. FŒRSTER and F. JORRE.

The authors describe a large number of experiments made with a view to the study of the formation of chlorates by the action of chlorine on the alkalis. The results of their researches agree generally with those of Lunge and Landolt (*Journ. Soc. Chim. Ind.*, 1885, p. 722), who studied the conditions under which the transformation of hypochlorite of lime in chlorate took place.

The classic equations which are supposed to represent the action of chlorine on caustic potash do not conform with the true facts of the case. When chlorine is passed through alkaline solutions, until they contain an excess, the chlorate is formed rapidly. The theory, according to which the chlorates should be formed more easily by passing the chlorine through warm concentrated solutions is incorrect. The addition of an acid to solutions of hypochlorite of varying strengths helps the formation of chlorates; the explanation of this fact is found in the theory of electrolytic dissociation.

It has also been found that, while solutions of hypochlorite and hypochlorous acid are stable when isolated, they, on the contrary, react on one another when mixed; the transformation into chlorate is rapid and complete.

As far as the electrolysis of solutions of chloride of lime containing chloride of potassium or sodium is concerned, the authors confirm the observations of Œttel; thick crusts, which gradually become detached, separate from the cathode, and free chlorine is given off.—*Journ. für Prakt. Chem.*, 1899, p. 53.

ON THE ANALYSIS OF MANUFACTURED INDIARUBBER.

By Dr. R. HENRIQUES.

During the years 1892, 1893, and 1894, I published the results of a research undertaken with the object of establishing analytical methods applicable to the treatment of manufactured indiarubber (*Moniteur Scientifique*, August, 1894, p. 617). The methods I then described have as a rule proved of practical value, not only in my own laboratory, but also in the hands of a certain number of colleagues employed in this industry. With practice I have since been able to simplify certain of these methods, and I now think the time has arrived for publishing the results obtained since the publication of my first papers.

I. *Estimation of the Total Sulphur and the Mineral Constituents.*

The method finally adopted is as follows:—A hemispherical porcelain crucible, about 6 c.m. in diameter, and of about 30 c.c. capacity, is covered with a watch-glass, allowing room for a glass stirring rod. This crucible is filled to about one-third, that is to say, with about 10 c.c., of pure concentrated nitric acid, and heated gently on a water-bath. One grm. of the finely divided sample is weighed out, and gradually added to the acid. The reaction soon commences with the disengagement of reddish fumes. This must be regulated by adding fresh portions of the substance in such quantities that the reaction keeps lively without getting too strong.

This can easily be done by either removing the crucible from the water-bath, re-heating, if necessary, and varying the quantity of the substance added. When the attack is finished the heating is continued until the disaggregation is complete. There is now no longer any loss to be feared by projection; the watch-glass can therefore be removed and wiped with a filter paper, which is placed in the crucible, and the evaporation is continued until the liquid takes a syrupy consistency. 20 c.c. of acid are then added, and the concentration repeated and carried as far as possible so as to drive off all excess of acid.

There now remains, to entirely destroy the organic matter. For this purpose we add to the syrupy extract a suitable dose of a mixture of dry soda (5 parts) and saltpetre (3 parts), and knead it well with the stirring rod so as to form an apparently dry powder. This is then covered with a layer of the mixture of soda and saltpetre, of which about 5 grms. is required altogether, and left on the water-bath until no more carbonic acid gas is given off. To prevent all chance of deflagration, which might

lead to losses, the calcination should be conducted very carefully. The crucible must be placed over a very small flame, which must be increased very gradually after some minutes, after having covered the crucible with another similar one so as to form a dome over it. If by chance the combustion becomes too fierce at any moment the projections will be stopped by the cover, and the substance thus caught must be melted apart, with a fresh dose of the oxidising mixture.

Under ordinary circumstances the mass gradually becomes black from the edges towards the centre, and only gives off empyreumatic fumes, which condense in the covering crucible, forming brown stains absolutely free from sulphur.

The first calcination once completed, the cover may be removed, and the substance heated to fusion, which can be accelerated by stirring with the glass rod. The entire operation of fusing requires from an hour to an hour and a-half. With a little practice it is easy to prevent any loss by deflagration. As a further precaution, it is as well, besides the careful regulation of the calcination, to use a very homogeneous mixture of soda and saltpetre, passed through a fine sieve, and thoroughly mixed with the nitric extract.

When the calcination is conducted without any deflagration, the same crucible can be made to serve for several operations. After cooling, the mass is taken up with boiling water and thrown on a filter. The whole of the sulphur in the sample will be found in the solution in the form of sulphate, and the metals remain on the filter in the form of oxides or carbonates, with the silica ; it is only when this latter is present in considerable quantities that a part of it will be found in the solution. The solution is acidulated with hydrochloric acid, evaporated to dryness on the water-bath, taken up with concentrated hydrochloric acid, the silica separated, and sulphate of barium precipitated in the usual manner.

The metallic oxides are taken up on the filter with warm hydrochloric acid, which leaves the silica only insoluble. In the hydrochloric solution the metallic oxides are estimated in the ordinary way.

When mercury is among the constituents of indiarubber —in the form of cinnabar, for example, it is often met with in dental rubbers—it must be estimated separately by attacking with nitric acid, diluting with water, filtering and precipitating with sulphuretted hydrogen.

Cinnabar is not such a rare constituent of indiarubber as might be supposed. Besides dental rubbers it is met with in certain fine hardened rubbers (stabilite), and in small quantities in many red rubbers, where it is associated with vermillion or golden sulphide of antimony, or even with oxide of iron, the colour of which it modifies to a certain extent.

II. *Estimation of Factitious Matter in Vulcanised Indiarubbers.*

The method previously described (*Moniteur Scientifique*, August, 1894, p. 617) has not been appreciably modified. Although it is rather long we have not succeeded in shortening the time occupied without interfering with its exactness. The only modification which has been introduced consists in the following, that instead of collecting the rubber exhausted with alcoholic potash on a tared filter, for the purpose of weighing by difference, we simply use any filter, from which the material becomes detached with the greatest ease during the drying in the oven. The complete desiccation is then effected separately and the weighing done direct.

III. *Estimation of Non-saponifiable Oils in Vulcanised Mixtures.*

The non-saponifiable oils, mineral oils, resin, and paraffin are often met with in commercial rubber, not only because the manufacturer adds a certain proportion to render the re-heating and cutting more easy, but also because several factitious rubbers and most regenerated or devulcanised rubbers contain them in notable proportions.

The estimation of these bodies is effected at the same time as that of the factitious matter. After treating the sample with alcoholic potash and weighing the residue, the latter is taken up by several applications of ether, which easily and completely dissolves all the non-saponifiable oils. The sample is simply ground with ether in a mortar and decanted, the rubber remaining well agglomerated. This extraction is continued as long as the solvent becomes coloured; the sample is then dried again, and the difference in weight is taken as non-saponifiable oil. It is on the residue from this extraction that the estimation of the combined sulphur and ash is effected, for the calculation of the approximate proportion of factitious matter present.

IV. *Estimation of Factitious Matter in Non-vulcanised Indiarubbers.*

The method of estimating factitious matter, such as already described (*loc. cit.*), gives very concordant results in the analysis of commercial vulcanised rubbers. It is applicable equally to the analysis of blocks or sheets of patent rubber, cut or otherwise, but it cannot be applied without modification to raw cut rubbers, that is to say, to mixtures prepared for vulcanisation.

Even after four hours boiling with alcoholic soda, the factitious matter is not entirely dissolved, and on repeating the extraction a fresh loss of weight is observed, which, however, does not even then correspond to the total amount of factitious matter contained in the mixture. As we already know that the factitious matter is entirely soluble in alcoholic soda and that the rubber itself is not soluble at all, this result can only be attributed to mechanical action, the thickness of the pieces preventing the interior layers from being reached by the solvent. To remove this difficulty I operated as follows:—

Five grms. of the material were placed in an Erlenmeyer flask, in contact with 25 c.c. of benzene. This was heated for an hour on a water-bath, after fitting a vertical condenser, and left to macerate for a night. The mass is by this time converted into a thick glue, to which is added 25 c.c. of normal alcoholic soda solution. After boiling again in the same manner for four hours, and then distilling off the solvents, the residue is taken up with boiling water. The whole is placed in a crucible, treated several times with water, and the coagulum of rubber separated. Filtering is useless, and the whole material is collected in a pasty mass, which is ground in a mortar with boiling water until all alkaline reaction disappears.

The proof that under these conditions the elimination of the factitious matter is complete, is furnished by the analysis of the remaining rubber, which does not contain a trace of sulphur. Further, a second extraction, conducted in the same manner, gives no supplementary loss of weight. The results of three analyses agree to within 1 per cent.

V. *Estimation of Non-saponifiable Oils in Non-vulcanised Rubbers.*

With non-vulcanised rubbers it is impossible to apply the method of estimation of non-saponifiable matter, described in paragraph III., on account of the partial solubility of the indiarubber itself in ether. In this case we make use of acetone, with which the rubber, freed from factitious matter and free sulphur, is boiled as in paragraphs II. and IV. The extraction by boiling is continued until the solvent is no longer coloured. The rubber then remains in a pasty gluey state, which is very difficult to dry for the purpose of weighing. Further, it is preferable to weigh the dissolved material direct; for this purpose the acetone extracts are filtered hot in a matrass, and after distilling off the acetone the residue is taken up with a little ether and left to evaporate spontaneously after filtration ; the residue is then dried in the oven at 100° and weighed.

Cold acetone does not easily dissolve the non-saponifiable substances met with in indiarubbers; but on boiling they are dissolved quite sufficiently to enable the total extraction to be accomplished without difficulty.

VI. *Estimation of Carbonic Acid.*

Carbonates are frequently used in the manufacture of indiarubber, particularly chalk and carbonate of magnesia, but we also occasionally meet with ceruse and carbonate of zinc (?). As, on the other hand, the oxides corresponding to these carbonates are often met with, it is interesting, in certain cases, to estimate the carbonic acid exactly. For this purpose a Geissler apparatus is used according to the directions given by Fresenius. It is essential that the rubber analysed should be of extreme fineness, and further—as vulcanised indiarubbers may contain sulphides, either through their being added, as in lithopone, or through their formation during the vulcanisation, by the action of sulphur on the metallic oxides—care must be taken that the carbonic acid gas given off does not carry with it any sulphuretted hydrogen.

For this purpose it suffices to replace the water with which the apparatus is charged, with a dilute solution of sulphate of copper. The carbonic acid gas which is then given off is entirely free from sulphuretted hydrogen.

This method, when used for the analysis of a non-vulcanised rubber, only gives bad results. In such a case, no matter how finely the sample may be divided, the hydrochloric acid is unable to reach the whole of the carbonate which is protected by the rubber; the attack, therefore, remains superficial. It is then necessary to have recourse to the preliminary extraction of the sample with nitrobenzene as proposed by O. Weber (Chemical News, Jan. 5, 1900, p. 5).

We operate on a few grms. of material which are boiled with nitrobenzene; the material is then washed with benzene, collected on a filter, dried, weighed, and an aliquot part taken for the estimation of the carbonic acid. The attack of the substance, which has now become pulverulent, is very rapid.

I might here remark that I have not, on many occasions, been able to obtain satisfactory results when following the method proposed by Weber, either in its original form or in the improved form which he has since devised. Non-vulcanised, or slightly vulcanised rubber, it is true, dissolves in boiling nitrobenzene, but if we have to deal with hard or highly charged rubbers it is very difficult to dissolve the whole of the gum, and the filtration of the residue presents insurmountable difficulties.—*Zeitschrift für Angewandte Chemie*, 1899, p. 802.

JUVENILE LECTURES.

At the Society of Arts, on Wednesday evening, 3rd inst., Mr. Herbert Jackson delivered the first lecture of his course addressed to a juvenile audience, the subject being "The Phenomena of Phosphorescence."

The lecturer commenced by a reference to the natural sources of phosphorescence as seen in the sea and in some cases of animal decay, &c. Phosphorescence and fluorescence might be treated as identical terms. They were illustrated by the exhibition of a phosphorescent jet of steam from a vessel containing water and phosphorus, and of the effect produced by sprinkling fluor-spar on a heated plate. He then proceeded to show how there were certain substances which showed light, generally of a yellowish or greenish colour, when exposed to the more refrangible rays,—the rays at the violet end of the spectrum,—and showed how the visible spectrum was apparently extended in length when the violet and ultra violet rays fell on a surface prepared with such materials as sulphate of quinine or thallene. On passing a slab of uranium glass along the spectrum, it was made manifest that no effect was produced on the glass by the red end, whereas as soon as it was placed in the violet rays it glowed with its characteristic greenish yellow colour. The phosphorescence of quinine and of fluorescein when the solution was dropped into a tall jar of water was also shown, and further illustrated by burning sulphur in oxygen in a jar immersed within a larger jar filled with the phosphorescent solution. The phosphorescence of such materials only lasted while the exciting cause was present, but there were other materials which continued to emit light after the exciting light had been withdrawn; of these possibly the best known was Balmain's luminous paint. A card coated with this was exhibited, and it was shown how the brightness of the phosphorescence was temporarily increased by heat and diminished by cold. It was, however, possible to make other similar substances which acted in the same way, but showed light of different colours, and Mr. Jackson exhibited a fine series of phosphorescent bodies, principally lime salts, which, when excited by the brilliant discharge from a large induction coil, glowed for a certain time with various tints, blue, yellowish green, and pinkish red. Phosphorescence could be produced in a block of lime, such as that used for the lime-light, but it was of very short duration. If, however, such a block were rapidly rotated in the close neighbourhood of the induction discharge, the phosphorescence produced by the discharge lasted long enough to be perceived on the side of the lime turned away from the spark as it rotated. After this the lecturer passed to the phosphorescent effects produced by the electrical discharge in vacuous tubes, and showed the very beautiful results in a long tube, in which a spark from an induction-coil was passed, as the air and the various gases with which the tube was filled were gradually exhausted.

NOTICES OF BOOKS.

The Cost of Living as Modified by Sanitary Science. By Ellen H. Richards. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1899. 124 pp. 12mo. Cloth.

Mrs. Robert H. Richards, Instructor in Sanitary Chemistry in the Massachusetts Institute of Technology, Boston, is already known to thoughtful housewives by her works on the "Chemistry of Cooking and Cleaning," and on "Food Materials and their Adulterations." In the present volume she discusses the prime economic problem of households, how to secure comfort, health, and rational pleasure for the family, to the greatest financial advantage, in the light of modern sanitary science. She writes especially for those who earn small salaries, say from fifteen hundred to three thousand dollars a year (£300 to £600), and whose station in life causes them to make greater expenditures than their incomes warrant; she believes that the problem is more difficult for them than for day labourers and artisans earning but six hundred dollars a year (£125), and whose aspirations are far smaller.

The book discusses Standards of Living, Household Expenditure, and its ideal division among certain departments; Food; Clothing in relation to Health; Emotional and Intellectual Life, and the Organisation of the Household.

Under Food the author makes an apt citation from a writer of antiquity: "Wherefore do you spend money for that which is not bread, and your labour for that which satisfieth not?" (Isaiah, lv., 2).

Mrs. Richards says: "Let not woman grasp for the reins of business until she can master the running of one home." That the household is not run on economic principles is acknowledged by all, and the author thinks that the "women's province is degraded by her own connivance, since knowledge is at her disposal and she does not avail herself of it."

This suggests the question how can such useful books as this one, for example, be brought to the attention of those housewives who most need them? The shiftless, ignorant, yet proud woman who thinks she knows it all, is the very one who can scarcely be induced to examine a thoughtful, economic treatise on the "Cost of Living," while the graduate of a woman's college, or it may be of a University, accustomed to serious study, will read, ponder, and profit. Mrs. Richards is, however, altruistic and thinks the outlook full of hope.

The volume is pleasantly written, free from technicalities, and can be easily "understanded of the common people." H. C. B.

Methods for the Analyses of Ores, Pig-Iron, and Steel; in use at the Laboratories of Iron and Steel-works in the Region about Pittsburg, Pa., Contributed by the Chemists in charge, and Edited by a Committee of the Chemical Section, Engineers' Society of Western Pennsylvania. Eastoe, Pa.: Chemical Publishing Co. 1898. Pp. 133.

The methods of analysis described in this volume were originally published in the year 1896, by the Engineers' Society of Western Pennsylvania, but, as the supply of copies became exhausted, the present publication, in a more convenient form, has been undertaken by the Chemical Publishing Company.

There are here described the methods used in sixteen different laboratories, and they will be of the greatest interest to all practical chemists engaged in this branch of chemical analysis. There is also an Appendix containing various special methods of analysis of ores and furnace products, the whole forming a valuable collection which will make a useful addition to the book-shelf.

Chemistry for Organised Schools of Science. By S. Parrish, B.Sc., A.R.C.S. (Lond.). With an Introduction by D. Forsyth, M.A., D.Sc. London: Macmillan and Co., Ltd. 1899. Pp. 262.

The method of treatment adopted in this book follows, to a great extent, that employed in the two years' course in Chemistry in the School of Science in the Leeds Higher Grade School, and the author has found that the order adopted is both reasonable and practical. We are glad to see that the balance is brought into practical use early in the course, instead of—as is so often the case—being left till towards the end. The fundamental principles and practice of chemical analysis can soon be acquired by an ordinarily intelligent student, and, in our opinion, the sooner he learns the use of the balance the better.

In the second year the greater proportion of the experiments are quantitative, while symbols, theory, and formulæ are introduced about the middle of the course.

The whole book seems to be well arranged and written, while the illustrations are numerous and instructive. We feel sure that students will find it of great service, as by its help much work can be done in the absence of a teacher. At the end of the book will be found a number of examination questions and exercises, and a good index.

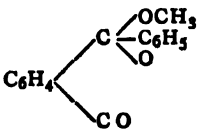
The Goldsmiths' Institute, New Cross, S.E.—Chairman of Governors: The Prime Warden of the Worshipful Company of Goldsmiths.—A course of fifteen lectures on Third Year Organic Chemistry will be given on Friday evenings, at 8.30, by William Jackson Pope, F.I.C., F.C.S., F.M.S., F.C.G.I., commencing on January 19th, 1900. In this course of lectures particular attention will be paid to the discussion of recent work and current views relating to organic chemistry. The lectures are suitable for advanced students preparing for the Organic Chemistry Examinations in Honours of the London University and Science and Art Department. Fee for the course, 5s.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxix., No. 26, December 26, 1899.

Researches on the Tautomerism of Benzoylbenzoic Acid.—A. Haller and A. Guyot.—Whatever the method used for the preparation of benzoylbenzoic acid the authors always obtain the same ether, crystallising in prisms melting at 52°. One of the methods employed excludes, to a great extent, the existence of a lactic form of the benzoylbenzoic compound. The conclusion is then drawn that the two forms of the ether do not exist, and that only the form $C_6H_4<^{COC_6H_5}_{COOCH_3}$ is susceptible of being prepared, the second form—



being unstable and quickly transposed into the other form.

The Temperature of Transformation of the Two Varieties of Mercuric Iodide.—D. Gernez.—The transformation of red quadratic mercuric iodide into orthorhombic yellow crystals, under the influence of heat, is a reversible reaction, but the phenomenon presents, in both cases, a retardation: (1) due to what may be called "crystalline surfusion," and which shows itself when the yellow iodide is slowly cooled to a lower temperature to that at which the transformation can be effected. This retardation may persist for some time. (2) The reverse of the last, the red crystals persisting at a temperature higher than that at which they can exist in the yellow variety. These phenomena explain the diversity of the various numbers previously given. The author discusses the theoretical explanation of the above two phenomena.

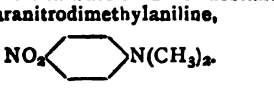
New Experiments on the Activity of Manganese with respect to the Phosphorescence of Strontium Sulphide.—José Rodriguez Mourelo.—In a previous paper the author described the result obtained in the preparation of phosphorescent strontium sulphide, by substituting, as the active material, bismuth subnitrate or manganese carbonate in very small quantities. The phosphorescence, though always intense, presents in this case a very clear yellowish green colouration. By varying the experimental conditions the author arrives at the conclusion that freshly prepared carbonate of manganese, precipitated and quite dry, and kept from contact with air, seems to possess the active power to the greatest degree.

Silicide of Molybdenum—E. Vigouroux.—Molybdenum prepared by M. Moissan's method in the electric furnace will unite directly with silicon. The substance formed, of which the composition is analogous to tungsten silicide, shows almost identically the same properties. It burns with incandescence in chlorine at about 300°, forming tetrachloride of silicon and black molybdenum perchloride which is deposited in the apparatus.

Molybdenum Bisulphide.—Marcel Guichard.—No previous method allows of the preparation of tolerably large quantities of pure molybdenum bisulphide. After several trials the author found two satisfactory preparations, the one giving crystalline and the other amorphous bisulphide. Both methods are described in detail and analysis given.

Action of Nitrous Acid on the Leucobase $C_{18}H_{24}N_2$.—A. Trillat.—Nitrous acid is allowed to act on an acidulated solution of the base $C_{18}H_{24}N_2$; a very

vigorous action then takes place. The compound resulting from this action is in the form of yellow needles, sometimes several c.c. long, which melt at 163°—164°. They are soluble in hot acetic acid, hydrochloric acid, and alcohol, but insoluble in water. This substance has been identified with paranitrodimethylaniline,



Heat of Neutralisation and Acidimetry of Cacodylic Acid.—Henri Imbert.—The author's experiments show that cacodylic acid is a feeble monobasic acid, and as it differs in reality from dimethylphosphoric acid, not only by the substitution of arsenic for phosphorus, but also by having two atoms of oxygen less. We see also that the presence or absence of oxygen in the molecule increases or diminishes the degree of energy of the acid function, since dialcoylphosphoric acids are acids which are strongly neutralisable by the same quantity of the base of helianthine and phthalein.

The Hydrate of Sodium Peroxide and the Preparation of Hydrogen Peroxide.—M. de Forcrand.—Vernon Harcourt discovered a crystallised hydrate of sodium, $Na_2O_2+8H_2O$, which was formed by the slow evaporation of aqueous solutions of Na_2O_2. The author continues this research, but finds a better method of preparation from a concentrated solution of Na_2O_2. The solution being made at 0°, and the temperature gradually raised to about 40°, on again cooling it to 0° an abundant crop of crystals separate out. No definite formula has been found for these crystals. The author also determined the heat of solution of this hydrate.

Anhydrous Sesquichlorides of Rhodium and Iridium.—E. Leidié.—The difficulty of preparing these substances by the direct action of chlorine on the metal has led the author to devise an indirect method for their preparation. Rh_2Cl_6 is most easily prepared by heating in a current of pure dry chlorine to 400° one of the three double chlorides of potassium, sodium, or ammonium, which have been dried at 100°—105°. Ir_2Cl_6 is obtained by heating the dry double salt $Ir_2Cl_6{\cdot}6NH_4Cl$ in chlorine gas to 440°.

Bulletin de la Société Chimique de Pari.
Series 3, Vol. xxi., No. 14.

General Method for the Synthesis of the Ketonic Acids.—E. E. Blaise.—None of the methods at present known lend themselves to the synthesis of the ketonic acids of the form $CH_3—CO—C(R)_2\ldots—CO_2H$. Now the dimethyllevulic and dimethylhexanonoïc acids, which are derived respectively from the oxidation of campholene and β-campholenic acid, belong to this type; the author has therefore sought for a new method for effecting their synthesis. The method adopted is based on the action of zinc-methyl on the chlorinised ethers of the bibasic acids,—

$$2COCl—C(R)_2\ldots—CO_2R+Zn(CH_3)_2= =ZnCl_2+2CH_3—CO—CR_2\ldots—CO_2R.$$

To obtain a pure chlorinised ether it is necessary to treat the corresponding ether acid with trichloride of phosphorus at the lowest possible temperature, and to be careful not to submit the product of the reaction to distillation, which would entirely change its composition.

A New Synthesis of Levulic Acid.—E. E. Blaise.—This synthesis has been effected by the action of zinc-methyl on the chlorinised succinic ether. The manner in which this reaction is carried out has a great influence on the return obtained. On the one hand, not more than half a molecule of the metallo-organic compound may be employed for each molecule of the chlorinised ether—

$$2\,\begin{matrix}CH_2—COCl\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}+Zn(CH_3)_2 = ZnCl_2+2\,\begin{matrix}CH_2—CO—CH_3\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}$$

Or, on the contrary, equal molecules may be taken and the reaction limited to its first phase,—

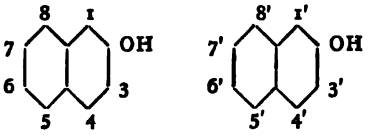
$$\begin{matrix}CH_2—COCl\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}+Zn(CH_3)_2= =\begin{matrix}CH_2—C(CH_3)(Cl)—O—Zn—CH_3\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}$$

the compound obtained being then decomposed by water,—

$$\begin{matrix}CH_2—C(CH_3)(Cl)—O—Zn—CH_3\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}+H_2O= =\begin{matrix}CH_2—CO—CH_3\\ |\\ CH_2—CO_2—C_2H_5\end{matrix}+CH_4+Zn(OH)(Cl)$$

Again, we can pour the zinc-methyl into the chlorinised ether, and proceed in the inverse sense. Experience has shown that the best returns are obtained by using equal molecules of chlorinised ether and zinc-methyl, and in pouring the first into the second, in such a manner that there will always be an excess of the metallo-organic derivative present. The author has also applied this method to dimethylsuccinic and dimethylglutaric acids for effecting the synthesis of dimethyllevulic and dimethylhexanonoïc acids,

The Constitution of β-Binaphtylol.—R. Fosse.—The number of binaphtylols foreseen thoretically is 28, the union of the two molecules taking place in the positions αα, ββ, or α-β. The binaphtylol obtained by the author is a derivative of α-α-binaphtyl; in fact, α-α-binaphtyl is obtained by distilling binaphtylol with zinc powder. If we call the carbon atoms of one molecule of naphtylol 1, 3, 4, ... 8, and the atoms of another molecule 1', 3', 4', ... 8',—



the junction can only take place between 1, 4, 5, 8, and 1', 4', 5', 8',—that is to say, the only possible junctions are

1 : 1'	1 : 4'	1 : 5'	1 : 8'
	4 : 4'	4 : 5'	4 : 8'
		5 : 5'	5 : 8'
			8 : 8'.

Action of the Aldehyds and the Aldehydic Chlorides on Binaphtylol Acetals.—R. Fosse.—The author has reacted with aldehydic chloride on sodic binaphtylolate with perfect success, the reaction being expressed thus:—

$$R—CH(Cl)(Cl)+\begin{matrix}NaO—C_{10}H_6\\ |\\ NaO—C_{10}H_6\end{matrix}= =2NaCl+R—CH(O—C_{10}H_6)(O—C_{10}H_6)$$

By an analogous method Tawildarow prepared ordinary acetal by reacting with bromide of ethylidene on ethylate of sodium.

On some New Derivatives of β-Binaphtylol.—R. Fosse.—The author describes the method of preparing and the composition and properties of several new derivatives of β-binaphtylol, among which are di-propionate of binaphtyl, $C_{26}H_{22}O_4$; di-butyrate of binaphtyl, $C_{28}H_{26}O_4$; phtalate of binaphtyl, $C_{28}H_{16}O_4$; bromobinaphtylol, $C_{20}H_{12}Br_2O_2$, &c.

On the Symmetrical Dinitro-ditolylcarbamides.—H. Vittenet.—The author has prepared the nitrated di-

tolyl carbamides by two methods, by which he has already previously obtained the substituted diphenylcarbamides: 1st, he reacted with the nitrotoluidines on $COCl_2$ in a 20 per cent solution in toluene, and 2nd, he reacted with the same bases on $CO<^{O-C_6H_5}_{O-C_6H_5}$. The method with phosgene gave good returns, but with carbonate of phenyl he got much lower returns and far less pure products. In this paper he fully describes these nitrotolyl carbamides, as well as the bases formed by their reduction.

Some Cuprous Phenylhydrazinic Salts.—J. Moitessier.—Phenylhydrazine forms, with cuprous chloride, bromide, and iodide, crystallised compounds analogous to those formed by ammonia, aniline, and pyridine. The cuprous phenylhydrazinic salts are almost insoluble in cold water, soluble in solutions of hyposulphite of soda, insoluble in alcohol and ether, which remove the phenylhydrazine. They gradually decompose in air, especially in moist air, as they also do very rapidly at a high temperature.

On the Aloïnes.—E. Léger.—The author states that all the aloïnes, with the exception of *nataloïn*, such as *socaloïn*, *barbaloïn*, *curacaloïn*, *sanaloïn*, have the same centesimal composition; the differences noticed are principally those of colour and the quantity of water of crystallisation contained in the aloïnes and their derivatives.

On Barbaloïn.—E. Léger.—The study of the derivatives of barbaloïn has confirmed the author's opinion that barbaloïn should be represented by the formula $C_{16}H_{16}O_7$, and that it contains 3OH.

The Solubility of the Proteoses and the Peptones in Alcohol.—Dr. J. Effront.—The author finds that the true peptone in the commercial article is soluble in alcohol, while the insoluble part, or that portion which is precipitated by sulphate of zinc, consists principally of the proteoses; he also establishes the point that the solubility of the proteoses depends on the reactions of the medium employed.

On the Estimation of Albumoses and Peptones.—Dr. J. Effront.—The author fully describes his method for the estimation of albumoses and peptones, which is based on the determination of the syntonin. In neutral solutions the albumoses are precipitated by alcohol, while the peptones go into solution.

On the Dissolving Power of Pepsine.—Dr. J. Effront.—Pepsine, like amylose, has a dissolving as well as a hydrating power. These distinct properties of pepsine have not yet been examined separately; the author has therefore thought it of interest to carry this out, and he here, in a rather long paper, gives the results of his experiments with acids, alkalis, chlorides, sulphates, and a number of other chemical substances; he finds than sulphates particularly have a very strong retarding action.

The Chemical Analysis of Rocks. Estimation of Potassium and Sodium.—E. Bonjean.—Already inserted in full.

MISCELLANEOUS

Cornell University Laboratory.—A new addition to the chemical laboratory at the Cornell University has recently been built at a cost of 55,000 dollars. It is intended for the accommodation of the divisions of Inorganic Chemistry and Physical Chemistry, and also to relieve the overcrowding in the old laboratory. The new building is 130 feet long by 65 feet wide, of most substantial build, and thoroughly equipped with apparatus and fittings of the highest class. The sub-basement, basement, and first floor are occupied by the division of Inorganic Chemistry, and the second floor by Physical Chemistry. At the end of the building on the basement floor is the assay laboratory, provided with seven crucible furnaces, five muffle furnaces, one gas assay furnace, and one powerful gas blast furnace; it is situated immediately above the ore-storage room; the adjoining rooms are arranged—one for gold and silver assaying, and one as a balance room. The last room is fitted with an electric furnace for chemical experiments where the electric arc is used as a source of heat. On the first floor there are—the private laboratory for the Professor of Chemistry, and two small rooms thoroughly equipped for research. These are each intended for the use of one student only. On the same floor are found the museum, the laboratory for spectroscopic work, and the lecture room. The whole of the second floor is devoted to physical chemistry, such as electro-chemistry, &c. The opportunities now offered by the Cornell University for the study of Physical Chemistry are, says Professor Bancroft, quite unequalled in the United States, and the equipment will bear comparison with that of the Leipsic laboratory, especially when one takes into account the fact that constant temperature rooms, spectroscopic laboratories, electric furnaces, &c., are available on the floors of the building assigned to inorganic chemistry.

MEETINGS FOR THE WEEK.

TUESDAY, 16th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 17th.—Society of Arts, 8. "Ventilation without Draughts," by Arthur Rigg.
—— Microscopical, 8. President's Address.
—— Institution of Mining and Metallurgy, 8. "A Development in Gravitation Stamp Mills," by D. B. Morison and D. A. Bremner. "Notes on Gold and Platinum Mining in the Ural Mountains," by D. A. Louis, F.I.C.

THURSDAY, 18th.—Chemical, 8. "Nitrogen Halogen Compounds," by Julius Stieglitz and E. E. Slosson. "Chlorine Derivatives of Pyridine—Part V., Synthesis of $\alpha\alpha'$-Dichlorpyridine and Constitution of Citrazinic Acid," by W. J. Sell, M.A., and F. W. Dootson, M.A. "Action of Fuming Nitric Acid on α-Dibromocamphor," by A. Lapworth, D.Sc., and E. M. Chapman. "Electrolysis of Nitrogen Hydrides and of Hydroxylamine," by E. C. Szarvasy, Ph.D.
—— Society of Arts, 4.30. "Our Work in India in the Nineteenth Century," by Sir William Lee-Warner, K.C.S.I., M.A. (This meeting will be held at the Imperial Institute).
—— Royal Institution, 3. "The Senses of Primitive Man," by W. H. R. Rivers, M.A., M.D., &c.

FRIDAY, 19th.—Royal Institution, 9. "Flight," by The Right Hon. Lord Rayleigh, M.A., D.C.L., LL.D., F.R.S.

SATURDAY, 20th.—Royal Institution, 3. "Neglected Byways in Music" (with Musical Illustrations), by Sir Hubert H. Parry, Mus. Doc., M.A., D.C.L.

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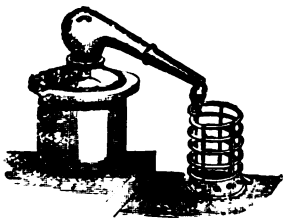

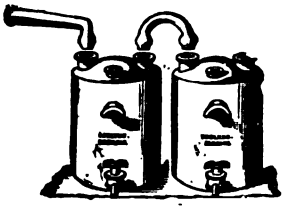

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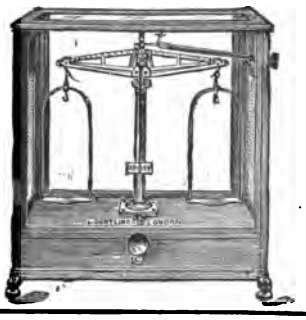

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THE CHEMICAL NEWS.

VOL. LXXXI., No. 2095.

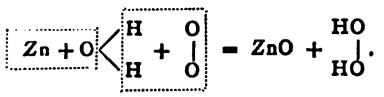
THE INFLUENCE OF WATER ON THE COMBUSTION OF CARBON MONOXIDE. A PLEA FOR "CONTACT ACTION."

By G. MARTIN.

BETWEEN the years 1882—1885 Traube showed in *all* cases of slow oxidation at ordinary temperature that—

1. In no case is there evidence that the atoms of the oxygen molecule are severed before oxidation proceeds (see *Note A*).
2. The atoms of the oxygen molecule are so firmly bound together that even substances like Na, K, S, C, &c., cannot sever the bonds between the oxygen atoms, and hence these substances remain unattached even when heated very intensely in DRY oxygen gas.
3. Absolutely pure water carefully freed from air has no action on metals such as zinc, iron, lead, &c.
4. But the moment *oxygen* and *water* are allowed to act *simultaneously* on the metal, oxidation proceeds, and hydrogen peroxide (H_2O_2) is in all cases formed.

He therefore regards oxidation as due to a decomposition of the water molecule under the contrary attractions of the metal for the oxygen of the water molecule and the free oxygen gas present for the hydrogen of the water molecule, thus :—

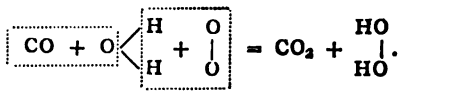


Notice that here the oxygen molecule does not break up, but combines with the hydrogen as a whole. It would hence appear that the conditions immediately preceding an oxidation at low temperature consist in a *complication*, not in a *simplification* or break down of the oxygen molecule as is usually believed.

In the above reaction, although the zinc tends by itself to decompose the water, yet the presence of a third body —oxygen—is necessary to determine the reaction. Indeed the oxygen here appears to act more by "contact" than anything else, and were it not that we can detect small quantities of H_2O_2 (which, however, are rapidly destroyed by side reactions), we should have a so-called "catalytic action" pure and simple. At higher temperatures, although the determining attractive power of the oxygen still remains, yet in consequence of the more violent motion of the atoms this attractive power is not sufficient to cause combination, but yet quite sufficient to ensure the progress of a chemical reaction.

It appears that Prof. Dixon has no right to neglect the effect of this "contact action" of oxygen in determining the course of a reaction, especially as Traube's experiments show that oxygen possesses sufficient attractive power to form H_2O_2. Although at higher temperature H_2O_2 may not be actually produced, yet the "tendency" for its production always remains, but is neutralised by the greater motion of the atoms (see *Note B*).

When Prof. Dixon, in 1884, discovered that *dry* carbon monoxide and oxygen gas will not explode, Traube, under the influence of his earlier experiments, put forward this theory to account for the action of water in rendering the CO and O_2 mixture inflammable (compare previous equation):—



(2). $CO + H_2O_2 = CO_2\ H_2O$.

Prof. Dixon has, I believe, succeeded in proving that Traube made some erroneous statements, but he has not yet proved that Traube is wrong in principle. Prof. Dixon explains the reaction by the following equations:—

(1). $CO+H_2O=CO_2+H_2$.

(2). $2H_2+O_2=2H_2O$.

Now Prof. Dixon bases his first equation upon the fact that *when CO* and *steam* are heated together, reaction (1) partially takes place.

He contradicted Traube's assumptions by means of experiments on the explosion of mixtures of carbon monoxide and hydrogen containing insufficient oxygen for complete combustion.

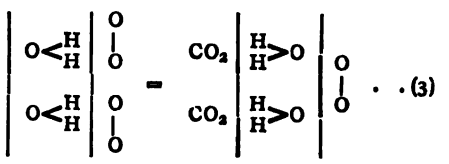
The case, then, is this:—Prof. Dixon states that *because* CO_2 and H_2 are formed when CO and H_2O are heated together; *therefore* when CO, H_2O, and O_2 are heated together, we also get hydrogen gas formed, which combines with free oxygen gas to form water, the oxygen having no independent effect in altering the character of the change.

I do not think Prof. Dixon is justified in assuming that experimental results deduced from one set of experiments under certain physical conditions—such, for instance, as a deficiency or absence of oxygen gas—apply to another set of experiments under totally different physical conditions—such as an excess or sufficiency of oxygen necessary for the complete combustion of the reacting materials, especially as Traube's experiments appear to indicate that oxygen exerts a certain "attractive force" on water which may totally change the course of a reaction.

The mere presence or absence of a substance may exert an enormous influence on the *character* or *nature* of the change.

A more probable view is that the explosion consists in a number of chemical reactions going on at one and the same time. As the relative masses of the reacting materials vary with the progress of the reaction, so too do the quantitative amounts of the several changes involved continually alter—one reaction predominating at one time, and another at another time, according to the varying amounts of the products of combustion present in the gas.

Thus at the beginning of the explosion, when there is an excess of steam and a sufficiency of oxygen, we probably get this reaction taking place (See Armstrong, "Presidential Address to Chem. Soc.," 1895).



When, however, the quantity of oxygen becomes more and more exhausted as the combustion proceeds, the reaction—

$$CO+H_2O=CO_2+H_2. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

comes more and more into evidence.

I do not mean to imply that these are the only reactions taking place. There would probably be intermediate reactions between (3) and (4).

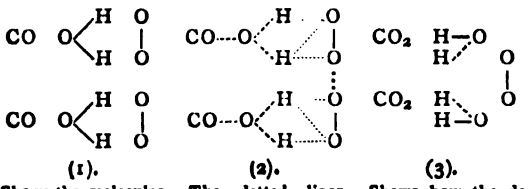
Now since we should expect (3) to proceed with less expenditure of energy than (4)—since in (3) the reaction

partakes of the nature of successive "link motion" between the molecules (see *Note C*), while in (4) we have an absolute disruption of the H_2O molecule, we should hence expect (3) to take place in the largest quantity, because it would proceed the most readily, but as the oxygen gas gets burnt up (4) should come more and more into evidence. (4) would probably proceed at a slower rate than (3), owing to the greater expenditure of energy necessary to bring it about.

If this view is correct a cinematographic series of photographs of the explosion should reveal a bright head of flame, followed by a tailing off of lesser intensity, or even waves, due to the different rates of propagation of the different reactions.

The tailing off should become more marked as the quantities of steam or oxygen present in the mixture diminish.

In (3) we can suppose with Traube that H_2O_2 first *tends* to be formed (see *Note D*), but that a more staple arrangement of the atoms immediately takes place thus:—



(1). Shows the molecules before they are in close proximity to each other.

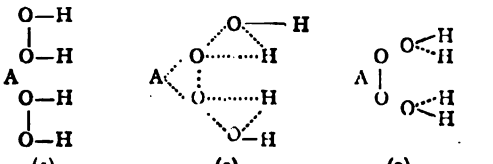
(2). The dotted lines show the new distribution of strain among the atoms when in close proximity to each other. The above does *not* represent a chemical compound, it represents a "condition."

(3). Shows how the decomposition proceeds under "contact" influence of the oxygen. It is the attraction of the oxygen both for the H and for itself that causes the decomposition to take place.

The change from (2) to (3) would be practically instantaneous, because it takes place *before* any H_2O_2 is *actually* produced. There is a *mere tendency* for the H_2O_2 to exist.

That such a mere *tendency* would have most important effects in determining chemical change cannot be doubted.

Contact action can thus be readily explained if we bear in mind the fact that molecules and atoms are in a state of *violent motion*. Thus we might have—



(1). Old distribution of strain.

(2). New distribution of strain.

(3). Decomposition under new strain.

The attraction of any substance A for the oxygen of the hydroxyl molecules being insufficient to cause union, but quite sufficient to cause a new distribution of strain among the atoms of the molecules.

Thus, as the centre of force shifted, the relative dependency of the atoms on each other would alter, and, considering that the atoms are in a state of violent motion, it is very easy to conceive the momentous importance such a momentary re-distribution of strain among the atoms would have in determining the course of chemical change.

Dixon's hypothesis has yet another fault. It is not *general* enough. Although at the time it was advanced it was capable of explaining the particular effect produced by water in determining chemical action in the *one* particular case of CO and O, yet later work has shown us that this particular case stands not alone, but is *one* only of an enormous number of cases where such an explanation as Dixon put forward is inadmissible, and a common solution must be extended to all.

How, for instance, can Prof. Dixon explain the fact that ammonium chloride does not dissociate in the absence of water, but sublimes unchanged?

On the "influence" theory here advanced it is readily explainable; the "pull" of the H_2O molecule on the already over-strained NH_4Cl molecule being sufficient to cause its disruption.

We are here reminded of the well-known phenomenon of "super-cooling." In this case we must suppose that when the substance is cooling below its solidifying point the presence of some foreign material by its "attractive" power alters the distribution of strain among neighbouring molecules, and hence alters the state of motion of their atoms and in consequence the substance quickly solidifies or changes its physical state.

In the case of NH_4Cl might not the H_2O molecule act mechanically like the foreign particles in the case of a supercooled liquid, and thus destroy the equilibrium of the over-strained NH_4Cl molecules? Once an ammonium chloride molecule has decomposed into NH_3 and HCl, the NH_3 and HCl act themselves like foreign particles and further the change, because part of their total energy is available for an external attraction.

The stability of the NH_4Cl molecule in this over-strained and abnormal condition might be explained as the stability of a system in motion—the momentum, so to speak, of the atoms in the molecule giving a certain amount of stability to the system. The presence of an external attraction (like that of a neighbouring H_2O molecule) might suffice to throw the atoms out of the grooves in which they run (speaking figuratively within the molecule, and hence cause a decomposition (see *Note D*), especially as the vapour is on the verge of decomposition. In the absence of such an external attraction there would be no reason why the state of motion within the molecule should not continue in virtue of its own inertia, and thus produce such phenomena as "super cooling", &c.

The reason why NH_4Cl molecules do not act like foreign particles towards each other, and thus mutually cause dissociation to take place, might be sought in the fact that the molecule has all its energy strained to the utmost in keeping together the tugging atoms, and hence has no energy left with which it can exercise an external attraction.

That water has an abnormal power of dissociating bodies in solution is well known, and is an argument for the above view.

Notes.

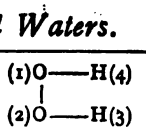
A.—Traube cited the following in proof of the fact that no "nascent oxygen" atoms are present during a "slow" oxidation:—(1) If "nascent oxygen" were produced by shaking up zinc with atmospheric oxygen and water (in which case H_2O_2 is produced), an easily oxidisable substance such as indigo sulphonic acid would quickly be oxidised and decolourised; but this is found not to be the case, although H_2O_2 is formed (abstracts *Chem. Soc.*, 1882, p. 795). (2). Zinc, when shaken up with air and NH_3, forms H_2O_2; but were the hypothetical active oxygen present, the NH_3 in the solution should be oxidised to NH_4NO_2 and NH_4NO_3. No traces of these substances could be detected. (3). When KNO_3 is reduced by zinc, no active oxygen atoms are produced, for the reduction to KNO_2 proceeds as readily in the presence as in the absence of air. The NH_3 produced in the reduction is *not* oxidised to ammonium nitrite or nitrate, even when there is a large excess of air present, and this although H_2O_2 can be detected. (4). When acidulated water is electrolysed, the platinum electrodes being separated by a cylinder of some porous material, Traube finds that no H_2O_2 is produced at the + pole (where nascent oxygen is liberated) but only at the − pole

where the oxygen gas dissolved in the water can come into contact with the nascent hydrogen as it is evolved. Now if the nascent oxygen combined with the water to form H_2O_2, we should certainly expect to find the H_2O_2 at the + pole, where the nascent oxygen is given off. Instead of this the H_2O_2 is only formed at the − pole, where the hydrogen is given off and can combine with the oxygen in the water about the − pole. In none of the above cases (and many others) could Traube detect the presence of active oxygen atoms, although at the same time oxidation of the water proceeded and also H_2O_2 was formed. It would appear then that a slow oxidation proceeds not by a break-down of the oxygen molecule into atoms, but rather by a complicated double reaction.

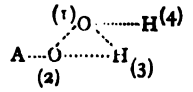
B.—At present a chemical attraction is only recognised when it is of sufficient strength to cause a chemical combination of the elements concerned. It cannot be doubted that many "chemical attractions" exist, but which are not evident because the attractive force is too feeble to cause an actual combination; nevertheless such a force might have important effects in determining the character of a reaction, although, indeed, no compound may actually be produced. This probably explains the so-called "catalytic" actions. Another point to be observed is that if a body exerts an attraction on another at some one temperature, it probably will still exercise an attraction at any other temperature—although the velocities of the atoms may so change that this constant attractive power will not exercise much control over the motion of the atoms—it is the motion that changes, not the attractive power.

C.—The energy absorbed by a "snapping" of one "bond" being immediately equalled or exceeded by the energy given out by the combination. If we imagine the molecules stretched in a chain, the attractions being equal on all sides of the constituent atoms, it is easy to see how an oscillatory movement of the atoms will propagate a chemical reaction with very little expenditure of energy.

D.—It may be conceived that a chemical reaction takes place in many cases by a preliminary association of the molecules together. This "association" need not necessarily represent an instantaneous "compound," it merely represents a "condition." During this period of association the relative "strains" among the atoms must shift, and thus prepare for a break up of the molecules, and as a result of the violent internal movements of the atoms in the molecules this "shifting of the strains" immediately intensifies the strain on some groups of atoms and weakens it in others, and hence we get groups of atoms detaching themselves to form new chemical compounds. Traube's work favour this view. (See also Dr. Armstrong's Presidential Address to the Chemical Society in 1895, where strong arguments are put forward for a preliminary "association" of molecules immediately preceding a chemical reaction). Now "contact action" is simply a case of this "association" of molecules, but in this case, although the force is sufficient to cause decomposition, yet it is not sufficient to cause combination as in the action of finely divided gold on the H_2O_2, &c., &c. To understand what is meant by the word "strain" among the atoms, the reader is referred to Mendeleeff's "Principles of Chemistry," vol. ii., Appendix i. (1897), where it is pointed out that we have strong reason (from spectroscopic and other physical and chemical work) to believe that the atoms of a molecule are in motion, and in consequence of this motion they exert a "pull" on each other. Now, by Newton's "Third Law of Motion," that "Action and Reaction are equal"; if an atom A exerts a force on an atom B, then B exerts an equal force on A. Contact action acquires a new significance if looked upon in the light of this conception. Consider, for example, the molecule—



whose atoms are numbered 1, 2, 3, and 4. According to the above the pull of each H atom on the neighbouring O atom is equal and opposite to the pull of the O atom on the H atom, and in turn the pulls of the O atoms on each other are equal, so that a state of dynamical equilibrium reigns throughout the molecule.



If now a substance A be brought up close to one of the O atoms (2), then owing to the presence of A the position of 2 will be altered in respect to 1, 3, and 4, and we should now expect that these atoms, 1, 3, and 4, would now shift their positions so as to exercise a joint pull on 2 equal and opposite *to the pull exerted on them by atom 2, part of the pull formerly expended on them by 2 now being transferred to* A. Now as these atoms shift their relative positions so as to automatically adjust themselves to the new state of equilibrium, it is easily understood if we bear in mind the internal motions of the atoms, how such a shifting might result in a decomposition of the molecule.

Merchant Venturers' Technical College,
Bristol.

THE

ESTIMATION OF AMMONIA, NITRIC ACID, AND NITROUS ACID IN NATURAL WATERS.

By L. N. WINKLER.

The estimation of the ammonia in natural waters is generally done by the colorimetric method, with the aid of Nessler's solution. The method followed consists in removing the calcic and magnesic salts from the water under examination, and adding to the clear liquid a certain quantity of Nessler's solution. In the presence of ammonia the mixture takes a yellowish tinge. A solution of chloride of ammonium of known strength is then added to exactly the same quantity of distilled water, free from ammonia, until the tint coincides exactly with that obtained with the natural water. When the Nessler's solution is dropped in to the distilled water, and the chloride of ammonium then added drop by drop, the former becomes cloudy, and can no longer be examined colorimetrically. Again, the typical solutions must be freshly prepared for each series of experiments, by mixing large quantities of water with small quantities of chloride of ammonium. I have found that by adding, besides the Nessler's solution, a solution of Seignette salt, or any other tartrate, the chloride of ammonium can be added without any precipitate being formed. The use of the Seignette salt is so advantageous that the elimination of the calcium and magnesium is superfluous; now it is exactly this factor which makes the colorimetric examination in question so long and difficult. We can, however, make a sufficiently exact estimation of the ammonia present in natural waters by adopting the following method. Two flasks are required, fitted with ground glass stoppers, and of a little more than 100 c.c. capacity; into one of these flasks we pour 100 c.c. of the water to be examined, and into the other 100 c.c. of distilled water free from ammonia; to each of these flasks are then added 2 or 3 c.c. of a solution of Seignette salt, saturated at the ordinary temperature, and a similar quantity of Nessler's solution. To the flask containing the distilled water we then add, drop by drop from a graduated burette, a solution of chloride of ammonium, of which each c.c.

contains a quantity of chloride equal to 0·1 m.grm. of ammonia. The flask is frequently shaken, and the addition of the chloride stopped when the distilled water has taken the same tint as the water under examination. The volume of the chloride of ammonium used enables us to calculate the amount of ammonia contained in 1 litre of the water. The solution of chloride of ammonium is prepared by dissolving 0·315 grm. of the salt in 1 litre of water; the best form of burette to use is one of Gay-Lussac's, of about 10 c.c. capacity, with an internal diameter of 6 or 8 m.m. The method described does not require the use of a colorimeter, and gives very exact results. The following are a few examples:—

Ammonia added to 1 litre.	Ammonia found in 1 litre.	Ammonia added to 1 litre.	Ammonia found in 1 litre.
M.grm.	M.grm.	M.grm.	M grm.
0·48	0·46	0·47	0·49
1·50	1·55	1·22	1·16
5·00	4·72	1·85	1·98

When the water to be examined is cloudy it must be filtered, using only a small filter, and rejecting the first 100 or 200 c.c.

The quantitative estimation of the nitric acid in natural waters is most easily effected by means of the brucine reaction (G. Lunge and A. Lucoff, *Zeitschr. fur Angew. Chem.*, 1894, No. 12).

When we mix one volume of a very dilute solution of a nitrate (1 : 100,000) with two volumes of concentrated sulphuric acid, and dissolve in this liquid, when cold, a small quantity of brucine, a beautiful rose-red colouration is produced. After standing for a considerable time the colour changes to an orange-yellow, and finally takes a citron-yellow tint; but on adding the brucine to the warm solution, the red colour produced remains for an instant only, and immediately gives place to the yellow tint. The intensity of the colour is in direct relation to the quantity of nitric acid present.

These facts enable us to estimate the quantity of nitric acid present in natural waters in a few minutes; the following is the method of procedure:—Two small flasks of 50 c.c. are used; into one of these we pour 10 c.c. of the water to be examined, and into the other 10 c.c. of distilled water. To each of these flasks is added 1 c.c. of a 2 per cent solution of sulphate of brucine, and then 20 c.c. of concentrated sulphuric acid. The nitrated water becomes yellow, while the distilled water remains colourless, that is, if the sulphuric acid is free from nitrates. We then add, drop by drop, to this latter solution, while still warm, a solution of potassic nitrate, of which each c.c. contains 0·1 m.grm. of nitric acid. After the addition of each drop the flask must be shaken, and the colour compared with that of the other one; when the tints agree the estimation is complete. The number of c.c. used, multiplied by 100, gives the quantity of nitric acid contained in 1 litre of the water under examination. When the water contains only traces of nitric acid, and very exact results are required, 50 c.c. of the water should be taken, and 100 c.c. of sulphuric acid.

The solution of potassic nitrate contains 0·187 grm. of the pure dry salt per litre; thus, each c.c. is equivalent to 0·1 m.grm. of anhydrous nitric acid, N_2O_5. The accuracy of the results obtained by this method is fairly good, as can be seen from the following figures:—

Nitric acid added to 1 litre.	Nitric acid found in 1 litre.	
	With 10 c.c. of water.	With 30 c.c. of water.
M.grms.	M.grms.	M.grms.
7·0	6·3	
2·0	2·5	2·0
13·0	11·8	13·0
0·5	0·9	0·7
21·0	21·7	
31·0	29·7	

It may reasonably be asked whether the organic matter contained in natural waters does not have an unfavourable influence on the results obtained. My experiments show that such is not the case. By this method I have estimated the nitric acid in a highly contaminated water, then in another portion of the same water I dissolved a small quantity of permanganate of potash, added a few drops of concentrated sulphuric acid, and heated on the water-bath for about half-an-hour. After having destroyed the excess of permanganate by means of oxalic acid, I estimated the nitric acid, and did not find any variation from the results first obtained. This is sufficient proof that the estimation of the nitric acid can be effected even in the presence of fairly large quantities of organic matter. When the water contains ferrous salts, they must be oxidised to the ferric state by means of permanganate. As nitrous acid acts on brucine in the same manner as nitric acid, it must be first oxidised to the state of nitric acid by permanganate, and deducted from the nitric acid found.

To detect nitrous acid in natural waters a small quantity of potassic iodide must be dissolved, then a little starch solution added, and a few drops of sulphuric acid. In the presence of nitrous acid, the iodide is set free and colours the starch blue. The reaction takes place according to the equation—

$$HNO_2+HI=NO+I+H_2O,$$

or $$N_2O_3+2HI=2NO+I_2+H_2O;$$

one molecule of N_2O_3 thus sets at liberty two atoms of iodine, so that, by titrating the iodine liberated with a solution of hyposulphite of soda, we can calculate the quantity of nitrous acid present. The titration of the iodine, however, has not a very definite termination, even if we use a larger amount of hyposulphite of soda than is theoretically required; the solution, which is decolourised with great difficulty, immediately returns to its original colour. This phenomenon is due to the presence of binoxide of nitrogen, which, as in the manufacture of sulphuric acid, here plays the important part of absorbing oxygen.

The protoxide of nitrogen combines with the dissolved oxygen, to form the binoxide, which sets iodine and hydriodic acid free, and is itself reduced to the state of protoxide. But the question bears a different complexion when the oxygen which interferes with the reaction is driven off. If we acidulate the water, and then add a carbonate or bicarbonate, the carbonic acid formed drives off not only the oxygen entangled in the water, but also the dissolved oxygen. On then dissolving iodide of potassium in the sample, it is perfectly easy to titrate the iodine set free without fearing the interference of the unfavourable factors which were just referred to. The estimation of nitrous acid may thus be effected in the following manner:—Take 100 c.c. of the water under examination; if it is cloudy or coloured yellow by ferric hydrate, it must be cleared by sedimentation and filtration. The sample taken is placed in a flask of 200 c.c. capacity with a long narrow neck, 20 c.c. of 10 per cent hydrochloric acid and 2 or 3 c.c. of starch solution are added. To this mixture is then gradually added 5 grms. of pure crystals of potassic bicarbonate, about 1 grm. at a time, waiting a little while after each addition, so that the froth does not run over. When the greater part of the salt (four-fifths) has been added, a small crystal of potassic iodide is thrown in, and then the remainder of the bicarbonate, so that the binoxide of nitrogen which is formed by the action of the nitrous acid on the hydriodic acid is driven out of the flask. The iodine set at liberty is titrated with a dilute solution of hyposulphite of soda, of which each c.c. contains 0·1 m.grm. of N_2O_3, a solution which can be prepared by diluting 26·3 c.c. of a decinormal solution to 1 litre. After titration the liquid should remain colourless for ten minutes; if it does not, it shows that the oxygen or the binoxide of nitrogen has been incompletely expelled.

The following figures show the great accuracy with

which even very small quantities of nitrous acid can be estimated:—0·406 grm. of nitrate of silver was dissolved in warm water, and an equal quantity of sodic chloride was added; on cooling, the cloudy liquid was made up to 1 litre and allowed to stand; the supernatant portion then constituted a solution of sodic nitrate containing 0·1 m.grm. of nitrous acid (N_2O_3) per c.c. The results obtained with different volumes of this solution are given below:—

N_2O_3 taken per litre.	N_2O_3 found per litre.	N_2O_3 taken per litre.	N_2O_3 found per litre.
1·00 m.grm.	0·96 m.grm.	1·00 m.grm.	0·95 m.grm.
3·00 ,,	3·00 ,,	3·00 ,,	3·00 ,,
5·00 ,,	4·95 ,,	5·00 ,,	5·11 ,,

It seemed of interest to make a few experiments with a view to finding out whether it is possible to estimate nitrous acid in the presence of nitric acid and ammonia. These two bodies were estimated by the method previously described; the results are given below.

Nitrous acid.		Ammonia.		Nitric acid.	
Taken per litre.	Found per litre.	Taken per litre.	Found per litre.	Taken per litre.	Found per litre.
M.grm.	M.grm.	M.grm.	M.grm.	M.grm.	M.grm.
0·4	0·34	0·4	0·42	3·0	2·9
1·4	1·32	2·4	2·00	5·0	5·2
3·0	3·03	4·0	3·59	14·0	13·5

I am therefore of the opinion that these results are most favourable to the method described, but I must once more repeat that the complete expulsion of the air is an essential condition to be observed—a condition that will be fully realised by following the directions given. I should also add that ferric salts also set iodine at liberty from a solution of hydriodic acid, but they can easily be removed by adding a solution of sodic hydrate, free from nitrite, to the water, and filtering. However, in natural waters the iron is nearly always found in the state of ferrous bicarbonate, and the ferrous salts have no action on iodides.—*Chemiker Zeitung*, 1899, No. 43, p. 454.

WORKS LABORATORY BURETTE RACK.

By C. SORDES ELLIS, A.I.C., F.C.S.

The instability of the ordinary burette stand is, I think, a fact well known to all chemical students. When college is left, and the chemist finds himself in a works laboratory, the bugbear of shaking burettes is still with him, and in an accentuated form.

To eliminate this inconvenience, and to greatly facilitate work, I have designed and had erected a rack, the end of which is shown in the accompanying diagram. Fig. 1 shows front view of a portion. W X and Y Z are two strips of wood, 1 inch by 2 inch and 5 feet long, with narrow edges to the front, held together at a distance apart of 18 inches by three pieces of wood (one only is shown, S S) morticed into the long pieces. The dotted lines A A A A are two shelves firmly fixed to the wall. To the shelves six strips of wood are screwed and morticed into the uprights (two are shown at S and S). The whole is made of pitch-pine, and fixed plumb at any convenient distance from the shelves behind and the bench beneath. In a second rack I had put up, I found that one support from top to middle upright, and two from bottom shelf to end ones, were quite sufficient to hold the whole firmly to the shelves. At convenient distances apart semi-circular depressions, G, are cut on the front of the frame, both top and bottom, in a perpendicular line.

In Fig. 2 will be seen details of the clip. It is made of 1/16 inch brass, and ½ inch or ⅝ inch wide. A square, K, is hinged at B to a longer piece, with a slight depression at G' and a slit at D. On the other side of G is another square of brass, with a slotted projection brazed on the back. A piece of brass rodding, E, is hinged at C so as to move forward; E has a thread cut on it which fits a

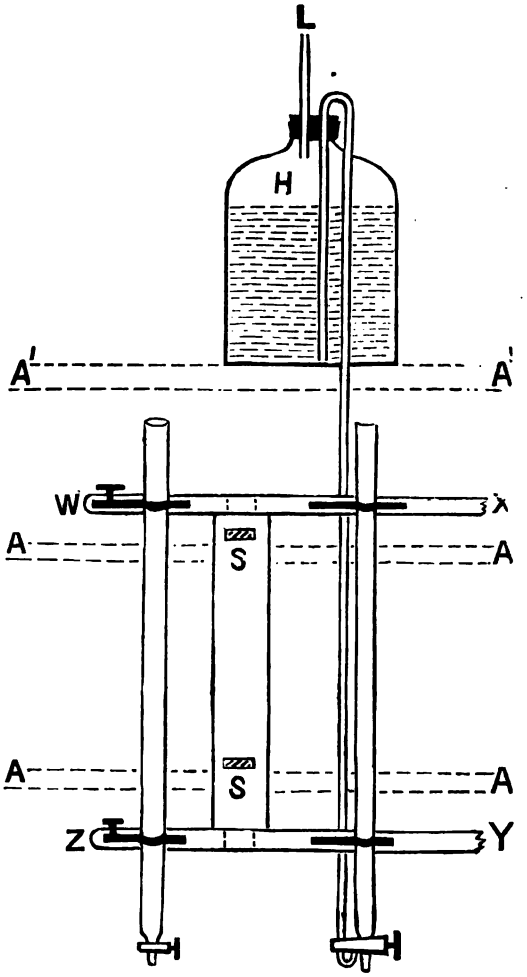


FIG I.

WORKS LABORATORY BURETTE RACK

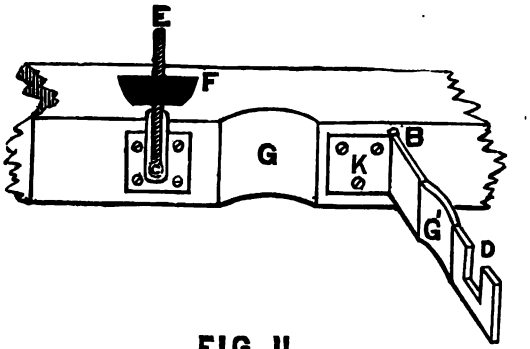


FIG II.

DETAILS OF CLIP & ATTACHMENT

milled edge nut, F; G and G' are both lined with thick baize or swansdown cloth. The whole is so arranged that when closed E will fall into the slit D, and on screwing up F the movable part will be brought up tight. The spring of the brass holds the burette firmly in position, perfectly rigid and perpendicular, being held by chips at top and bottom. If the rack is made to the dimensions given above, it will be found to take six burettes comfortably.

I have found the arrangement shown in the second burette, Fig. 1, most handy when large quantities of a standard solution are in constant use. The bottle, H, on a shelf, A' A', is fitted with a cork, through which two holes have been bored; into one a short piece of glass tubing is inserted, and the end drawn out to a very fine point; through the other hole a long syphon tube is placed, which is joined up to the burette having a two-way tap.

ON THE

VOLUMETRIC ESTIMATION OF CERIUM.*

By PHILIP E. BROWNING.

SOME forty years ago Bunsen (*Annalen d. Chem. u. Phar.*, cv., 49) showed that the oxide obtained by the ignition of cerium oxalate might be estimated volumetrically by bringing it in contact with potassium iodide and strong hydrochloric acid, and determining the iodine set free. This method may be briefly described by a translation of part of the original article:—"The substance to be determined is weighed out in a glass flask of from 10 to 15 c.c. capacity, a few crystals of potassium iodide are added, and the neck of the flask is drawn out by the aid of a blowpipe to a narrow opening. The flask is filled almost to the narrowing of the neck with hydrochloric acid which is free from chlorine or iron chloride, and a little sodium carbonate is added in order to displace the last trace of air by carbon dioxide. The flask is then closed by sealing off the neck in the blowpipe, and warmed in a water-bath until the cerium compound is completely dissolved, and the quantity of iodine set free is determined by iodometric analysis."

The anhydrous dioxide prepared by the ignition of the oxalate or hydroxide is very slowly acted on by acids, especially when pure (Rose, *Handbuch der Analytischen Chemie*, Band. i., 219). For this reason the method which Bunsen described has remained the only one adapted to the satisfactory volumetric estimation of the ignited dioxide.

Two portions of the dioxide were prepared by treating the crude cerium chloride in concentrated solution with gaseous hydrochloric acid (Dennis and Magee, *Zeitschr. f. Anorgan. Chem.*, iii., 260) to saturation to remove the iron. The cerium chloride was then dissolved in water, potassium hydroxide added in excess, and chlorine gas passed until the precipitate became distinctly orange in colour and the solution gave a strong odour of chlorine (Mosander, *Phil. Mag.*, xxviii., 241; Dennis, *Zeit. f. Anorgan. Chem.*, vii., 252). This operation was repeated until a portion of the precipitate dissolved in acid showed no didymium absorption bands when examined before the spectroscope. The whole precipitate of the dioxide was then dissolved in hydrochloric acid, and the oxalate precipitated by ammonium oxalate in large excess. The precipitated oxalate was then washed thoroughly with hot water until the washings gave no test for hydrochloric or oxalic acids and ignited to the dioxide. Another portion of the dioxide was later prepared by precipitating a solution of pure cerium chloride by means of ammonium oxalate, washing and igniting as described. The dioxide in all three cases was of a light chamois colour, and uniform results were obtained from the three portions.

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, 1899, vol. viii., No. 48.

A Modification of the Method of Bunsen (with G. A. HANFORD and F. J. HALL).

Weighed portions of the pure cerium dioxide were placed in small glass stoppered bottles of about 100 c.m.³ capacity, together with a grm. of potassium iodide free from iodate and a few drops of water to dissolve the iodide. A current of carbon dioxide was passed into the bottle for about five minutes to expel the air, 10 c.m.³ of pure strong hydrochloric acid were added, the stopper inserted, and the bottle heated gently upon a steam radiator for about one hour until the dioxide dissolved completely and the iodine was set free. After cooling the bottle, to prevent loss of iodine upon removing the stopper, the contents were carefully washed into about 400 c.m.³ of water, and titrated with standard sodium thiosulphate to determine the amount of iodine liberated according to the well-known reaction—

$$2CeO_2+8HCl+2KI=2CeCl_3+2KCl+4H_2O+I_2.$$

A few blank determinations were carried through in the bottles without the presence of the cerium dioxide to determine the amount of iodine set free under these conditions. The amount obtained was uniformly equal to 0·04 c.m.³ of the 1/10 N iodine solution which was taken as the correction and applied to all the determinations. The results follow in Table I.

TABLE I.

	CeO_2 taken. Grm.	CeO_2 found. Grm.	Error. Grm.
1.	0·1000	0·0994	0·0006 −
2.	0·1032	0·1034	0·0002 +
3.	0·1016	0·1017	0·0001 +
4.	0·1054	0·1041	0·0013 −
5.	0·2010	0·2021	0·0011 +
6.	0·1104	0·1109	0·0005 +
7.	0·1914	0·1907	0·0007 −
8.	0·1604	0·1603	0·0001 −
9.	0·2146	0·2145	0·0001 −
10.	0·1108	0·1099	0·0009 −
11.	0·1346	0·1347	0·0001 +
12.	0·1540	0·1534	0·0006 −
13.	0·1976	0·1968	0·0008 −
14.	0·1230	0·1240	0·0010 +
15.	0·1199	0·1201	0·0003 +
16.	0·1524	0·1528	0·0004 +
17.	0·1212	0·1211	0·0001 −
18.	0·1528	0·1543	0·0015 +

In order to obtain a further check upon the accuracy of the method, portions of the cerium dioxide were weighed out and placed in a distillation apparatus previously employed for similar purposes and described in former articles from this laboratory, viz., a Voit flask, serving as a retort, sealed to the inlet tube of a Drexel wash-bottle, used as a receiver, the outlet tube of which was trapped by sealing on Will and Varrentrapp absorption bulbs. In the retort the cerium dioxide together with 15 c.m.³ of water, 1 grm. of potassium iodide, and 10 c.m.³ of pure strong hydrochloric acid, were placed. In the receiver were 100 c.m.³ of water and 2 to 3 grms. of potassium iodide, and in the bulbs a dilute solution of potassium iodide. Before adding the hydrochloric acid a current of carbon dioxide was passed through the apparatus for some minutes. After adding the acid, the liquid was boiled in the current of carbon dioxide* to a volume of 15 c.m.³, when the free iodine had almost completely left the retort and passed into the receiver, and the apparatus was allowed to cool.

The iodine in the receiver was titrated directly with sodium thiosulphate, and that in the retort after dilution of the residue to about 400 c.m.³, the later amount seldom exceeding the equivalent of a few drops of 1/10 N iodine solution.

* The carbon dioxide gas was furnished by a Kipp generator from marble and hydrochloric acid of one-half strength, both of which had been boiled previously to remove all air.

The results follow in Table II.

Here also blank determinations were made, but no correction was found to be necessary.

An attempt early in the work to titrate by an alkaline arsenite the iodine liberated, after neutralising the hydrochloric acid, brought out some curious results which seem worthy of mention.

TABLE II.

	CeO_2 taken. Grm.	CeO_2 found. Grm.	Error. Grm.
1.	0·1028	0·1013	0·0015 –
2.	0·2060	0·2055	0·0005 –
3.	0·2014	0·2012	0·0002 –
4.	0·1716	0·1711	0·0005 –
5.	0·0974	0·0972	0·0002 –
6.	0·1600	0·1587	0·0013 –
7.	0·1268	0·1254	0·0014 –
8.	0·1276	0·1268	0·0008 –
9.	0·1620	0·1612	0·0008 –
10.	0·1016	0·1011	0·0005 –
11.	0·1548	0·1543	0·0005 –
12.	0·1352	0·1342	0·0010 –

In these experiments the contents of the bottles after the cerium had dissolved were carefully washed into a Drexel wash-bottle, upon the inlet tube of which was fused a thistle tube with a stopcock, and to the outlet tube a Will and Varrentrapp absorption trap. In the trap a solution of potassium iodide was placed, and through the thistle tube a saturated solution of potassium bicarbonate was added to complete neutralisation of the acid. Any iodine carried mechanically by the carbon dioxide should be held by the potassium iodide solution in the trap. After neutralisation the free iodine was titrated by standard arsenious oxide solution. The results appear in Table III.

TABLE III.

	CeO_2 taken. Grm.	CeO_2 found. Grm.	Error. Grm.	
1.	0·1000	0·0987	0·0013 –	I.
2.	0·1005	0·0981	0·0024 –	
3.	0·1030	0·1009	0·0021 –	
4.	0·1500	0·1475	0·0025 –	
5.	0·1030	0·1005	0·0025 –	II.
6.	0·1010	0·0988	0·0022 –	
7.	0·1510	0·1508	0·0002 –	
8.	0·1530	0·1485	0·0045 –	
9.	0·2045	0·2011	0·0034 –	
10.	0·2000	0·1958	0·0042 –	
11.	0·1334	0·1302	0·0032 –	III.
12.	0·1354	0·1330	0·0024 –	
13.	0·1312	0·1294	0·0018 –	
14.	0·1308	0·1277	0·0031 –	
15.	0·1060	0·1042	0·0018 –	
16.	0·1602	0·1567	0·0035 –	
17.	0·1504	0·1488	0·0016 –	

As will be seen by the table, an average error of about 2 per cent runs through the entire set. The natural conclusion would be that the cerium dioxide contained some impurity; but, as the first, second, and third samples, very carefully prepared, gave the same results, it seemed necessary to look elsewhere for an explanation. Two possible causes suggested themselves: first, mechanical loss during the process of neutralisation, and second, the possible formation, under the conditions, of iodine chloride, which if formed would in the process of neutralisation probably take the form of potassium chloride, iodide, and iodate, and thus some of the originally free iodine would be withdrawn from the amount titrated. To test these theories, portions of the 1/10 N iodine solution roughly equivalent to the amounts of iodine set free by 0·1 and 0·2 grm. of CeO_2, were drawn off into bottles previously filled with carbon dioxide, treated with the usual amount of strong hydrochloric acid (10 c.m.³), and, after standing from thirty to forty-five minutes, neutralised and titrated as already described. The results were most interesting, and seemed to show a loss of iodine closely equivalent to that shown by the results of Table III., and proportional to the amount of iodine originally present. A few determinations were carried through in the same way, except that the neutralisation was omitted and dilution and titration with thiosulphate substituted. These showed a loss of iodine well within the limits of such a process. The results follow in Table IV.

TABLE IV.

With Arsenious Oxide.

	Iodine 1/10 N taken. C.m.³.	Iodine 1/10 found. C.m.³.	Error. C.m.³.	Equivalent error on CeO_2. Grm.	
1.	5·22	5·07	0·15 –	0·0026 –	
2.	5·09	4·97	0·12 –	0·0021 –	
3.	5·10	4·97	0·13 –	0·0022 –	
4.	5·66	5·47	0·19 –	0·0033 –	
5.	5·10	4·97	0·13 –	0·0022 –	
6.	10·22	9·97	0·25 –	0·0043 –	
7.	10·21	9·97	0·24 –	0·0041 –	

With Sodium Thiosulphate.

1.	5·	5·01	0·01 +	0·0002 +	
2.	5·	4·99	0·01 –	0·0002 –	
3.	10·	10·02	0·02 +	0·0003 +	
4.	10·08	10·12	0·04 +	0·0007 +	

(To be continued).

THE

IODOMETRIC DETERMINATION OF GOLD.*

By F. A. GOOCH and FREDERICK H. MORLEY.

In a recent attempt to measure small amounts of gold in solution by titrating with sodium thiosulphate the iodine set free in the action of an excess of potassium iodide upon auric chloride, Peterson (*Zeit. fur Anorg. Chem.*, xix., 63) has been led to conclude that, on the average, one-half more thiosulphate is used up in changing the characteristic starch iodide blue to the faint rose colour which precedes entire bleaching than is called for upon the theory that the thiosulphate is simply converted to the tetrathionate in the usual manner. Peterson explains the anomaly upon the hypothesis that, besides acting upon the free iodine, the thiosulphate is used up coincidently by interaction with the aurous salt, formed in the reduction, with formation of a gold sodium thiosulphate on the type of the well-known silver sodium thiosulphate. The reaction of this hypothesis is in the nature of things most improbable, since there is no reason to suppose that the soluble double thiosulphate could resist the action of the free iodine which is present to the end—the appearance of the rose colour,—and our study of the reaction of sodium thiosulphate upon the mixture of gold chloride and potassium iodide, the account of which follows, discloses no evidence of the consumption of more thiosulphate than is demanded by the usual theory, which postulates the simple formation of the tetrathionate by the interaction of the thiosulphate and free iodine.

It appeared in the course of our preliminary experimentation that, while practically similar results were obtained by adding the thiosulphate until the blue of the starch iodide had changed to rose, the indications were somewhat more concordant when the final rose colour was developed by adding iodine to the solution from which the blue had been bleached to colorlessness by a slight excess of the thiosulphate.

It appeared, also, that the reduction of the auric salt,

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, October, 1899.

with the consequent liberation of iodine, is conditioned by the volume of the solution, the mass of the iodine present, and the time of action.

The following statement, in which each result is the average of several titrations in close agreement, shows the effect upon the immediate evolution of iodine brought about by adding varying amounts of water to the gold solution before introducing the iodide, and the effect of different amounts of iodide at different dilutions.

	Potassium iodide.					Gold chloride.	Volume before the addition of the thiosulphate.
Grm.	.. 0·01	0·02	0·05	0·1	0·2	0·00087	C.m.
Sodium thiosulphate, nearly N/100. c.m.s.	0·81	0·81	0·81	0·82	0·84	,,	15
	0·77	0·78	0·80	0·81	0·81	,,	25
	0·74	0·72	0·78	0·79	0·80	,,	50
	0·61	0·61	0·68	0·76	0·79	,,	100
	0·45	0·49	0·60	0·72	0·75	,,	200

It is evident that for the smaller amounts of iodide the liberation of iodine decreases rapidly with the dilution. The larger amounts at the highest concentration show readings a trifle above the normal—perhaps because the well-known effect of concentrated solutions of a soluble iodide upon the delicacy of the starch end-color begins to appear. At volumes lying between the limit of 25 c.m.^{3} and 50 c.m.^{3}, 0·1 grm. of potassium iodide is an appropriate amount to use; at a volume of 15 c.m.^{3}, 0·01 grm. to 0·05 grm. of the iodide will do the work; and at lower dilutions, as will appear in the tabular statements to follow, even less of the iodide is effective.

In the series of experiments of which the details are given in Table I., use was made of a solution of pure gold chloride containing 0·8710 grm. to the litre—as determined by careful precipitation in the usual manner by ferrous sulphate, and by an alkaline solution of formaldehyde, according to the method of Vanino (*Ber. D. Chem. Gesellsch.*, xxxi., 1763). A nearly centinormal solution of iodine was prepared by diluting to a litre 100 c.m.^{3} of nearly decinormal iodine in potassium iodide carefully standardised against exactly decinormal arsenious acid. A nearly centinormal solution of sodium thiosulphate (containing 1·7012 grm. of $Na_2S_2O_3$ to the litre) was made by diluting to a litre 100 c.m.^{3} of a nearly decinormal solution of that reagent which had been standardised carefully against the standard iodine prepared as described. The solution of potassium iodide employed contained 10 grms. of that salt in the litre.

In conducting the experiments, a convenient amount of the solution of gold chloride was drawn from a burette, potassium iodide was introduced in the amounts indicated (always several times the theoretical equivalent of the gold, and more than enough to dissolve the aurous iodide precipitated at first), a sufficiency of clear starch indicator was added, the starch blue was bleached by the thiosulphate, and the iodine was added until the liquid assumed a faint rose colour. Upon the theory that potassium iodide sets free two atoms of iodine for every molecule of auric chloride (or every atom of gold) present, and that the thiosulphate acts only upon the free iodine to form the tetrathionate in the usual manner, every c.c. of the thiosulphate solution used in the reaction after deducting the amount equivalent to the iodine introduced to get the end-colour, should represent—

$$\frac{197{\cdot}3}{2(158{\cdot}22)} \times 0{\cdot}0017012 = 0{\cdot}001061 \text{ grm. of gold.}$$

Plainly, these results accord reasonably with the theory that two molecules of the thiosulphate are the equivalent in this reaction of two atoms of iodine and one atom of gold. There is no evidence whatever of the excessive action affirmed by Peterson.

The strength of the standard solutions used in the experiments described was such that an error of 0·01 c.m.^{3} in reading the volumes used would correspond to an error of 0·00001 grm. of gold. It is not to be expected that such readings can be trusted ordinarily to a higher degree of accuracy than 0·02 c.m.^{3}. In case all three solutions

TABLE I.

Gold chloride .. .. .. = 0·8710 to 1 litre.
Sodium thiosulphate, nearly N/100 .. .. = 1·7012 ,, ,,
Iodine, nearly N/100 .. .. = 1·3697 ,, ,,
(Volume at beginning of titration approximately 50 c.m.^{3}).

	$AuCl_3$ taken.	KI taken.	$Na_2S_2O_3$ used.	Gold found.	Theory for gold.	Error.	Per cent.
	C.m.^{3}.	Grm.	C.m.^{3}.	Grm.	Grm.	Grm.	
1.	5	0·05	4·02	0·00426	0·00435	−0·00009	2·1
2.	5	0·05	4·01	0·00425	0·00435	−0·00010	2·3
3.	5	0·05	4·06	0·00431	0·00435	−0·00004	0·9
4.	5	0·05	4·07	0·00432	0·00435	−0·00003	0·7
5.	5	0·05	4·04	0·00428	0·00435	−0·00007	1·6
6.	10	0·08	8·17	0·00867	0·00871	−0·00004	0·5
7.	10	0·08	8·15	0·00864	0·00871	−0·00007	0·8
8.	10	0·08	8·16	0·00865	0·00871	−0·00006	0·7
9.	10	0·08	8·15	0·00864	0·00871	−0·00007	0·8
10.	10	0·08	8·19	0·00869	0·00871	−0·00002	0·2
11.	10	0·08	8·46	0·00897	0·00871	+0·00026	3·0
12.	10	0·08	8·24	0·00874	0·00871	+0·00003	0·3

TABLE II.

A.

Gold chloride .. .. .. = 0·0871 to 1 litre.
Sodium thiosulphate, nearly N/100 .. .. = 1·7012 ,, ,,
Iodine, nearly N/100 .. .. = 1·3697 ,, ,,
(Solution of gold chloride not diluted before mixing with potassium iodide).

	$AuCl_3$ taken.	KI taken.	$Na_2S_2O_3$ used.	Gold taken.	Gold found.	Error.
	C.m.^{3}.	Grm.	C.m.^{3}.	Grm.	Grm.	Grm.
1.	10	0·01	0·83	0·00087	0·00088	+0·00001
2.	10	0·01	0·83	0·00087	0·00088	+0·00001
3.	10	0·01	0·80	0·00087	0·00085	−0·00002
4.	10	0·02	0·84	0·00087	0·00089	+0·00002
5.	10	0·02	0·88	0·00087	0·00093	+0·00006
6.	10	0·02	0·82	0·00087	0·00087	0·00000
7.	10	0·02	0·88	0·00087	0·00093	+0·00006
8.	10	0·02	0·83	0·00087	0·00088	+0·00001
9.	10	0·10	0·80	0·00087	0·00085	−0·00002
10.	10	0·10	0·82	0·00087	0·00087	0·00000
11.	10	0·01	0·83	0·00087	0·00088	+0·00001
12.	9	0·01	0·73	0·00078	0·00077	−0·00001
13.	8	0·01	0·65	0·00070	0·00069	−0·00001
14.	7	0·01	0·58	0·00061	0·00061	0·00000
15.	6	0·008	0·51	0·00052	0·00054	+0·00002
16.	5	0·008	0·41	0·00043	0·00044	+0·00001
17.	4	0·005	0·35	0·00035	0·00037	+0·00002
18.	3	0·005	0·24	0·00026	0·00026	0·00000
19.	2	0·003	0·21	0·00017	0·00022	+0·00005
20.	1	0·003	0·10	0·00009	0·00011	+0·00002

B.

Gold chloride .. .. .. = 0·0871 to 1 litre.
Sodium thiosulphate, nearly N/1000 .. .. = 0·17012 ,, ,,
Iodine, nearly N/1000 .. .. = 0·13697 ,, ,,

21.	10	0·01	8·39	0·000871	0·000890	+0·000019
22.	9	0·01	7·45	0·000784	0·000790	+0·000006
23.	8	0·01	6·30	0·000697	0·000668	−0·000029
24.	7	0·008	5·50	0·000610	0·000583	−0·000027
25.	6	0·008	5·12	0·000523	0·000543	+0·000020
26.	5	0·005	4·23	0·000435	0·000449	+0·000014
27.	4	0·005	3·38	0·000348	0·000358	+0·000010
28.	3	0·003	2·55	0·000261	0·000270	+0·000009
29.	2	0·003	1·71	0·000174	0·000181	+0·000007
30.	1	0·003	0·90	0·000087	0·000095	+0·000008

should be read to this limit of accuracy with the errors of all lying in the same direction, the summation of errors would correspond to 0·00006 grm. of gold.

In the experiments in Table II., therefore, solutions obtained by properly diluting those of the previous series were employed. The use of a more dilute solution of gold obviated the necessity for diluting the mixture of gold chloride and the iodide before titrating with the thiosulphate. It was found, however, that when the N/1000 solution of iodine is employed a correction of 0·1 c.m.³ for volumes not exceeding 30 c.m.³ becomes necessary—the amount required to bring out the rose colour in blank tests containing no gold. After the introduction of 0·1 c.m.³ of N/1000 iodine into a mixture of potassium iodide and starch indicator of volume not exceeding 30 c.m.³, a single drop of the gold solution—equivalent to 0·000002 grm. of gold—gave a distinct rose colour; before such adjustment of the solution five drops—equivalent to 0·000010 grm. of gold—were needed to develop the same colour.

These results run on the whole as regularly as could be expected, and the use of the dilute standard solutions is obviously of advantage.

In the practical application of any such process for the determination of gold, the elementary form of that metal is the natural starting-point. To get the metal into solution with chlorine water or mixed hydrochloric and nitric acids is an easy matter, but the removal of the excess of the oxidiser by evaporation without reducing some auric chloride to the aurous form is difficult. We have found, however, that the free chlorine may be removed from a solution of auric chloride, without reducing the auric salt, by treatment of the solution with ammonia in excess, boiling gently, acidifying with hydrochloric acid, and heating if necessary to re-dissolve the precipitate by ammonia, again treating with ammonia and heating, and once more acidifying. On the second addition of ammonia no precipitation usually takes place with the amounts of gold which we have thus handled, perhaps because enough ammonium chloride has been found to hold it up.

The following table contains determinations made with such a solution of pure gold leaf—tested gravimetrically as to purity.

Table III.

Gold chloride made by dissolving 0·0104 grm. of pure gold in the manner described and diluting to 200 c.m.³.

Sodium thiosulphate, nearly N/1000 .. .. = 0·17012 to 1 litre.
Iodine, nearly N/1000 .. = 0·13697 „ „
Potassium iodide .. .. = 10 grms. „ „

(Portions were treated with the potassium iodide without previous dilution).

	$AuCl_3$ taken. C.m.³.	KI taken. Grm.	$Na_2S_2O_3$ used. C.m.³.	Gold taken. Grm.	Gold found. Grm.	Error. Grm.
1.	1	0·005	0·55	0·000052	0·000058	+0·000006
2.	1	0·005	0·55	0·000052	0·000058	+0·000006
3.	2	0·005	1·06	0·000104	0·000112	+0·000008
4.	2	0·005	1·08	0·000104	0·000114	+0·000010
5.	5	0·01	2·45	0·000260	0·000260	0·000000
6.	5	0·01	2·50	0·000260	0·000265	+0·000005
7.	5	0·01	2·45	0·000260	0·000260	0·000000
8.	5	0·01	2·50	0·000260	0·000265	+0·000005
9.	5	0·01	2·50	0·000260	0·000265	+0·000005
10.	10	0·02	4·86	0·000520	0·000515	−0·000005
11.	10	0·02	4·85	0·000520	0·000517	−0·000003
12.	10	0·02	4·90	0·000520	0·000520	0·000000
13.	10	0·02	4·80	0·000520	0·000512	−0·000008
14.	10	0·02	4·84	0·000520	0·000516	−0·000004

Obviously, this method, which rests upon the hypothesis that sodium thiosulphate acts in the normal manner only upon the iodine set free by the interaction of gold chloride and potassium iodide, offers trustworthy means for the determination of small amounts of gold.

NOTICES OF BOOKS.

A Short History of the Progress of Scientific Chemistry in our own Times. By W. A. Tilden, D.Sc., F.R.S., Professor of Chemistry in the Royal College of Science, London. London: Longmans, Green, and Co. 1899. Pp. 276.

The volume now before us is the outcome of a series of "Lectures to Working Men" given by Prof. Tilden in the sixtieth year of the Queen's reign. He took as his subject the progress made in the science and practice of chemistry during the preceding sixty years, and he showed how the present system of chemistry has been built up from the previously existing order of things.

At the commencement of the Queen's reign, Liebig's influence and teaching was beginning to be felt, and the new organic chemistry was attracting attention.

The author first deals with matter and energy, and draws attention to the two important principles, viz., the conservation of matter and the indestructibility of energy, and the work done by Newton, Rumford, Davy, Joule, Boyle, &c., on these subjects. The chemical elements, their distribution in nature and recognition by the chemist, are next discussed. The recognition of an element does not necessarily imply, as is often supposed, the isolation of the element itself. Fluor-spar, for instance, was known as far back as the middle of the last century to yield an acid analogous to hydrochloric acid, but the element fluorine was not isolated until Moissan achieved this result in 1886. In the same way alumina was known as a distinct earth in 1754, but the metal was not isolated until Wöhler obtained it in 1828. Since the year 1837, when only 54 elements were known, there has been, and still is, a steady augmentation of the number recognised as such, and in 1897 their number was nearly 80.

The atomic theory was first applied to chemistry by Dalton, but he was obliged to admit later on that his simple hypothesis of single atoms joining to form compound bodies was not applicable to all cases, and was opposed to well-established facts; it served, however, as a starting point for the now fully-developed theory.

In Chapter V. the author considers the origin and development of the ideas of valency and the linking of atoms.

The first important step in this direction was made by Gerhardt, and adopted by Laurent in 1842. According to this system the formula of each substance represents that quantity of it which, in the state of vapour, would occupy two volumes, the unit volume being the space occupied by one part by weight of hydrogen gas at standard temperature and pressure. The next important step was taken by Würtz in 1849, when, while studying the action of potash on cyanic and cyanuric ethers, he was led to the discovery of the compound ammonias; Würtz regarded ammonia as a link between inorganic and organic chemistry. The development of synthetical chemistry is dealt with in Chapter VI. This subject has made remarkable strides of late years, and, as the author points out, has now reached the borderland where chemistry and physiology meet.

Stereo-chemistry (Chapter VII.), or chemistry in three dimension, is founded on the ideas expressed by Pasteur in general terms, though the original discovery of right and left-handed polarisation was made by the French physicist, Biot. The fundamental idea of stereo-chemistry, however, arises immediately out of the doctrine of atoms. In this subject the names of van 't Hoff, Le Bel, Kekulé, and others are well known. Chapter IX. is devoted to

discoveries relating to the liquefaction of gases, a subject sufficiently fresh in our memories not to require more than a passing notice.

We must congratulate the author on the excellent manner in which he has reviewed the subject of the progress of chemistry, a subject so vast as to cause considerable hesitation as to what to take and what to leave; he has, however, steered the middle course, and while not giving much time to the older and exploded doctrines, he has pointed out the influence they have had on our more modern and fully-developed ideas, the greatest of which is the atomic theory, without which organic chemistry would simply be a mass of confusion.

Introduction to Chemical Technical Analysis. By Prof. F. Ulzer and Dr. A. Fraenkel. Authorised translation, with appendix by the translator, Hermann Fleck, Nat. Sc.D. Illustrated. Philadelphia: P. Blakiston, Son, & Co. London: Kegan Paul, Trench, Trübner, & Co., Ltd. Pp. 188.

The value of manipulative skill cannot be over-estimated in chemical analysis; it is therefore important to give students as wide an experience as possible, with special instruction in carrying out the methods employed in the analysis of industrial products. The authors of this book have had much experience in teaching technical chemical analysis, and have therefore been able to give many methods of this description in a series of examples selected from a variety of products.

It is assumed that the student is already fairly well qualified as an analyst, all the examples given in this volume being special methods for individual cases. They include products of and materials used in technical chemistry, such as pyrites, sulphuric acid, brine, hydrochloric acid, soda, Weldon mud, furnace gases, &c.; cement and clay, the raw materials for the products of which are limestone, marl, and clay; the metallurgical industry; alloys and fertilisers; all of these being examples of inorganic analysis.

In organic analysis we find examples in the sugar industries; fermentation; fats, waxes, and oils; mordants and tanning materials; textile and dyeing industries; and the products of the coal-tar industry.

In the appendix, which has been added by the translator, there are a few special methods given for the analysis of white paint (white-lead), manganese dioxide, bleaching lime, &c.; asphaltes and food stuffs, such as milk, butter, flour, tea, &c.

We must call special attention to a note on page 7, in which it is stated that in all temperatures C = Celsius, unless otherwise indicated. We think this is unfortunate, as C is always understood to mean centigrade in England, Celsius being practically unused, and not conveying much meaning to the majority of chemists. The book is well printed, and should be a useful addition to the library of the commercial analyst.

CORRESPONDENCE.

RELATIONS BETWEEN THE ATOMIC WEIGHTS AND PHYSICAL PROPERTIES OF ELEMENTS.

To the Editor of the Chemical News.

Sir,—When the apparent equal expansion of gases was first observed, it was thought that if 273 volumes of a gas were taken at 0° C., at −273° C. there would be no volumes of gas, or at any rate they would be in a state in which there was no molecular activity. I have, therefore, written absolute zero, understanding an, at present, theoretical state when all molecular activity has ceased.

Mr. Bayley, in his letter of last week, takes exception to my "assigning a positive value to zero in one instance or the other." But consider the case of any two elements, say hydrogen and iron. In the state which is to us normal, hydrogen is a gas and iron a solid. To get hydrogen into the solid state we have already to apply to it several hundreds of degrees of cold. If then we take these two solids and wish to get them to the state of absolute zero, we must in each case lower the temperature by a range in some way proportional to the atomic weights. So that in the case of these two elements we have—

Absolute zero for iron ∝ atomic weight.

Absolute zero for hydrogen ∝ atomic weight + number of degrees necessary to reduce it to the same molecular state as the iron; or generally

Absolute zero = $(c \times \text{at. wt.}) + n$;

where n is the number of degrees necessary to reduce the element to the same molecular state as the "scratch" element.

Mr. Bayley further remarks that "it must not be overlooked that boiling-points, unless taken in an absolute vacuum, must be arbitrary." That any boiling-point could have a positive value I would not assert, but if the boiling-points under discussion were all taken under the same pressure the results can be compared with fairness.

To speak of taking a boiling-point in absolute vacuum must be a slip of the pen on Mr. Bayley's part, as by the definition of a boiling point, one such point under these conditions would be impossible, for by the term absolute I understand that the vapour given off by the substance itself is also withdrawn.

This he has himself shown in his paper of the same issue on "The Relation between Boiling-point and Melting-point in the Hydrocarbons," adding that if a substance in absolute vacuum be placed near a source of heat the temperature will rise in a certain way. This might be so if it could be explained how heat may be transmitted through an absolute vacuum; and even then the boiling-point is not dependent on the temperature, but on the pressure. If an absolute vacuum could be obtained, a lump of iron placed therein would boil, according to the definition. Thus, even if heat could be transmitted through an absolute vacuum, what Mr. Bayley terms "intrinsic boiling-point" concerns the intermolecular state, but not the boiling-point.—I am, &c.,

Alfred Wertheimer.

Stirling Chemical Works, Stratford, E.
January 9th, 1900.

PHYSICAL EQUILIBRIUM OF MOLECULAR SYSTEMS.

To the Editor of the Chemical News.

Sir,—In the address of Sir W. Roberts-Austen, delivered before the members of the Iron and Steel Institute, on May 4th, 1899, the following words occur:—"We must not lose sight of these relations of carbon and iron which involve physical equilibrium. Even the astonishing association of iron and carbon monoxide, in the volatile gaseous compound, with which the distinguished name of Mond is connected, affords a veritable triumph for dynamical chemistry. It is generally supposed that ozone is dissociated at 160° C., but Dewar has devised a beautiful experiment to prove that ozone has *two centres of stability*, and one of these is near the melting-point of platinum (*vide* "Year-book" of Royal Institution of Great Britain, 1887-9, p. 559). It seems to be the same with the relationship of hydrogen and iron. We have recently learned that iron and hydrogen appear to be completely dissociated at 800° C., and yet the same iron heated to a

still higher temperature, say 1200° C., will still yield hydrogen."

Silicon hexchloride (Si_2Cl_6) is, I believe, another example of this "double centre of stability" alluded to in the above.

According to the Text-books, "Silicon hexchloride (Si_2Cl_6), a colourless liquid which boils at 146° C., has a vapour which is perfectly staple below 350° C.; beyond that temperature the molecule gradually breaks down, until at 800° C. the compound is completely decomposed. If, however, the temperature of the vapour be suddenly raised from below 350° to above 1000° C., curiously enough we find no decomposition has taken place." We have, therefore, to deal with a compound staple below 350° and above 1000° C., but yet incapable of existing between these temperatures.

This cannot be explained by the hypothesis advanced by Dewar to account for the formation of ozone from heated oxygen gas, namely, the dissociation of the oxygen molecule into atoms, which unite on cooling to form ozone.

Can any of your readers give any other instances of similar phenomena, or advance any theory to account for the behaviour of silicon hexcloride?

The following occurred to me as a possible explanation:—

Let us assume that the molecule of a staple vapour is a system in a state of harmonious motion, and not a mass of atoms in discord, or, in other words, that the molecule is held together more by a certain correlation or harmony in the times of vibration of the atoms than by any very strong internal atomic attractions. It may easily be conceived that as the temperature increases, the mode of motion of some of the atoms in the molecule gradually becomes incompatible with that of neighbouring atoms, and thus discord and jarring is introduced. The atoms in the molecule no longer stir clear of each other.

In a word the atoms are now "Out of Time." Consequently the molecule becomes unstaple and breaks up. At a still higher temperature the motion of the atoms in the molecule may be so altered that there again exists a harmony in their periods or modes of vibration. The atoms then cease to hinder and check each other, and thus settle down in a state of harmonious motion and consequent staple equilibrium. The atoms are now again "In time" and the molecule is staple.—I am, &c.,

GEOFFREY MARTIN.

13, Hampton Road, Bristol.
January 7th, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

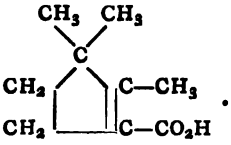
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 1, January 2, 1900.

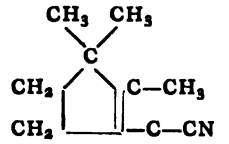
Manganic Oxidation of Citric and Malic Acids.—G. Denigès. — When a cold solution of citric acid is treated with potassium permanganate, the mixture becomes brown very rapidly with effervescence, and then gradually becomes colourless. The gas escaping is CO, showing that the point of least resistance of the citric acid is attacked. HO—C—CO.OH is transformed by oxidation into the ketonic group CO, with the simultanous evolution of CO and water. To identify this compound the author utilises the property which the ketone has of giving with mercuric sulphate a compound insoluble in water. In the case of malic acid, when this acid is treated hot with a sufficient quantity of $KMnO_4$, in addition to the decolorisation of the reagent, ordinary aldehyde is evolved, recognised by its odour. However, if the permanganate is added cautiously and not in excess, the decolouration is produced with no evolution of CO_2. In this case only the group CH.OH is attacked and transformed into CO, so as to produce oxalacetic acid, $HO.CO—CH_2—CO—CO·OH$. This acid can be recognised by the property it possesses of forming an insoluble mercury compound, not with the sulphate but with the acetate.

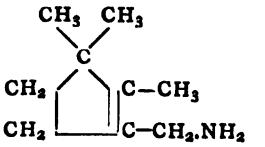
Acidimetry.—Henri Imbert.—In a previous paper the author mentioned that phenolphthalein admits of showing a feeble acidity, but a strong basicity, and inversely heliantine, a strong acidity and a weak basicity. He now extends his researches by means of these two indicators, adopting the following order:—Acidimetry of (1) phenols, (2) monobasic fatty and aromatic acids, (3) halogenated monobasic acids, (4) nitrated monobasic acids, (5) alcohols or phenols of monobasic acids, (6) pluriphenolic monobasic acids, (7) amines of monobasic acids.

Amines enclosing the Camphor Radicle.—G. Blanc.—The comparative study of isolauronolic and β-campholenic acids, and chiefly the consideration of their products of oxidation, shows clearly that there exists between them the same relation as that which exists between benzoic acid and phenylacetic acid.



If this relation is true it is easily seen that the partial reduction of isolauronolic nitrite ought to give a base identical with β-amino campholene.





However, this reduction does not easily take place. The author investigates the various substances formed.

Allotropy of Benzophenone. — M. Œchsner de Coninck.—Benzophenone presents two allotropic modifications, as was shown by Th. Zincke, but little is known about the conditions of transformation of the stable into the unstable variety. The author's researches show that, if he has not allotropised benzophenone by dissolving it twice in alcoholised water, slow oxidation, prolonged a sufficient time, should be stated amongst the conditions which allow of the transformation of the stable into the unstable variety of benzophenone.

Bulletin des Travaux de la Société de Pharmacie de Bordeaux, September and October, 1899.

These two numbers contain no matter of chemical interest.

November, 1899.

A New Method for the Detection and Estimation of Small Quantities of Iodoform.—G. Denigès.—The author found that by heating an ammonia compound of the aromatic series with iodoform, he was able, thanks to the easy dissociation of this latter body under the influence of heat, to join the amine both to the methanic group $H—C\leqq$ and the acid radical I, necessary to form a salt of rosaniline, of which the intensity of the colouration enabled him to detect traces only of iodoform. He tried a large number of aromatic ammonia compounds, and obtained better results with the substituted amines in

amidogen than when the substitution took place in the nucleus. In practice two cases may present themselves; 1. The iodoform may be separated by ether from the substances with which it is mixed. 2. The iodoform may be in a solvent miscible with ether, and cannot therefore be isolated by its help. In the first case the ether is evaporated in a crucible, and 3 or 4 drops of dimethylamine, which was found to give the best results, added; if there is even only a trace of iodoform present, the mixture immediately takes a very distinct yellow colour. The liquid thus obtained is placed in a small test-tube, and heated nearly to boiling for a few moments only; on cooling, a little alcohol is added, and a liquid is obtained which appears red by transmitted and violet by reflected light if much iodoform is present, and violet only when there is only a small quantity of iodoform. In the second case 4 or 5 drops of the material—iodoformed gaïacol for instance—are put in a tube with an equal quantity of dimethylamine; the mixture takes a yellow colour, the intensity of which is in proportion to the quantity of iodoform present; on carefully heating the mixture and diluting with alcohol the violet colour is produced.

Contribution to the Study of the Products of the Oxidation of Morphine.—A. Loubiou.—Having occasion last year to prepare some oxydimorphine, the author tried the method of Polstorff and Brokmann; this method being so slow, and the difficulty of purification being so great, he was led to try and devise a more rapid method. He tried some experiments with peroxide of hydrogen and binoxide of lead, and obtained a very white and well-crystallised product with the former. The return by this new process is less than that with ferricyanide, but the product is obtained pure immediately; further, the operation does not take more than an hour to perform.

MEETINGS FOR THE WEEK.

MONDAY, 22nd.—Society of Arts, 8. (Cantor Lectures). "The Nature and Yield of Metalliferous Deposits," by Bennett H. Brough.

TUESDAY, 23rd.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 24th.—Society of Arts, 8. "Local Government and its relation to Parish Water Supply and Sewerage," by W. O. E. Meade-King, M.Inst.C.E.

THURSDAY, 25th.—Royal Institution, 3. "The Senses of Primitive Man," by W. H. R. Rivers, M.A., M.D., &c.

FRIDAY, 26th.—Royal Institution, 9. "Motive Power—High Speed Navigation—Steam Turbines," by Charles H. Parsons, M.A., F.R.S., M.Inst.C.E.

—— Physical, 5. "Some Developments in the use of Price's Guard Wire in Insulation Tests," by Prof. Ayrton and Mr. Mather. "Reflection and Transmission of Electric Waves along Wires," by Dr. E. Barton and L. Lownds. "The Frequency of the Transverse Vibrations of a Stretched India-rubber Cord," by T. J. Barker.

SATURDAY, 27th.—Royal Institution, 3. "Neglected Byways in Music" (with Musical Illustrations), by Sir Hubert H. Parry, Mus. Doc., M.A., D.C.L.

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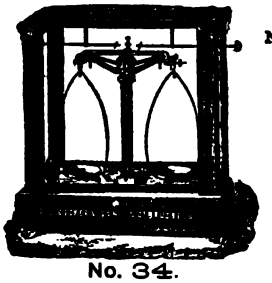

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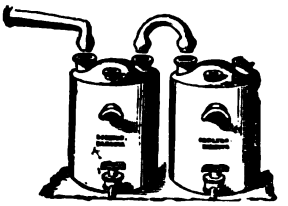

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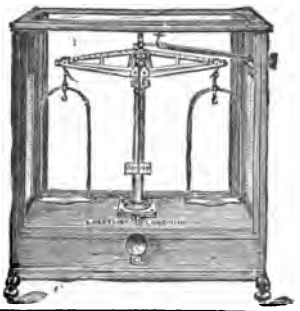

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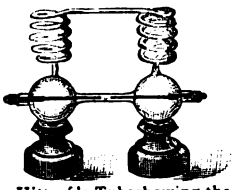

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Vol. LXXXI., No. 2096.

THE ESTIMATION OF INDIGO BLUE AND INDIGO RED IN NATURAL AND SYNTHETICAL INDIGOS.

By W. F. KOPPESCHAAR.

Of all the methods used for the estimation of indigo, the colorimetric method is the best, but only when the material contains no other colouring matter. In the opposite case, the solution obtained by treatment with concentrated sulphuric acid, diluting, and filtering, has a dirty appearance when contrasted with the pure solution of indigotine in the colorimeter.

In the method of titration by permanganate, we can never be certain that the impurities have not been oxidised at least partially. Further, the exact termination of the reaction is difficult to observe, and finally, if the sample contains a notable proportion of indigo red, the method is no longer applicable.

In the same way, in the determination of the oxygen necessary to oxidise a solution of white indigo prepared by reduction by means of hydrosulphite of soda, ferrous sulphate, grape sugar, &c., we cannot be certain whether the indigo is completely dissolved or whether the other substances do not also absorb oxygen.

As a consequence of the new methods of production, indigo (as well from Java as that from India) now contains more indigo red than it did formerly. Synthetical indigo red is now generally employed, but it is not identical with natural indigo red, and contains about 25 per cent of indigotine. The carmine from synthetic indigo red is easily soluble in concentrated solutions of chloride of sodium, while that from natural indigo is practically insoluble.

The method proposed by the author for the estimation of indigo blue and indigo red depends on the solubility of red and brown indigo in glacial acetic acid; blue indigo is insoluble in this reagent.

0·5 grm. of the finely-powdered sample is heated on the water-bath with 100 c.c. of glacial acetic for about an hour, in a conical flask of 8 or 9 c.m. diameter; the flask is then left in an inclined position, the liquid just reaching the neck, until all the insoluble matter is deposited. The clear liquid is then poured on an asbestos filter, prepared by placing a quantity of coarsely ground pumice-stone in a glass funnel up to 1 c.m. of the top, and covering this with a layer of asbestos. Care must be taken in decanting to leave the greater part of the insoluble residue at the bottom of the flask. The intensity of the colouration of the filtered liquid shows immediately whether the sample contains much or little indigo red.

In the latter case, a fresh quantity of acetic acid is added to the contents of the flask, and, after standing, decanted on the same filter. If, on the contrary, the sample is poor in indigo red, the small quantity that remains in the flask will not interfere with the ultimate estimation, and the washing may be omitted.

The asbestos and the pumice-stone, which shows whether any indigo blue has passed over, are removed from the funnel. This latter is placed stem upwards on the flask, so that its contents fall into the flask, and it is finally washed with 50 c.c. of pure sulphuric acid. After heating for two hours at 70°, and shaking frequently, the indigo blue is completely sulphonised. The solution is then poured into a 250 c.c. flask, and filtered after having been made up to that volume with water: 25 c.c. of the filtrate are diluted to 500 c.c., and the indigo blue determined colorimetrically by comparison with a solution containing 0·1 grm. of pure indigotine per litre. This pure indigotine may be prepared by digesting pure indigo with acetic acid on a filter of hard paper and drying.

To estimate the indigo red, 5, 10, or 25 c.c. of the acetic solution is neutralised with caustic soda. For 5 c.c. we require 12 c.c. of a 20 per cent solution of soda. After standing for fifteen minutes, the precipitate is collected on a small filter and washed with a 5 per cent solution of soda to remove the indigo brown, then with 2 per cent acetic acid to neutralise the soda with which the filter is impregnated. After drying, the filter is shaken up with glacial acetic acid in a 50 c.c. flask and the colour of the solution is compared with that of a solution containing 0·05 grm. of pure indigo red in 1 litre of glacial acetic acid.

The pure indigo red is obtained from indigo, and purified first by the method described above, and then by sublimation at reduced pressure.

For the estimation of indigo red in the synthetic product containing indigo blue, only 0·1 grm. is taken. The accuracy of this method depends on the sight of the operator. The observations made by the author showed differences varying from 0·2 to 0·8 per cent with indigo blue, and much smaller ones with indigo red.—*Zeitschr. Anal. Chem.*, 1899, xxxviii., [1], p. 1.

A METHOD FOR THE ESTIMATION OF FERROCYANIDES IN OLD PURIFYING MATERIALS.

By E. DONATH and B. M. MARGOSCHES.

The estimation of ferrocyanides is an operation which has often to be done by commercial chemists engaged either in the manufacture of prussiate both by the old process from residues rich in nitrogen and potash, a process which is gradually being abandoned, and by the new process which is now being adopted, the starting-point of which consists of exhausted purifying materials.

Some time ago Zulkowsky (*Dingl. Polyt. Journ.*, 1883, 249, 68) described a volumetric method for estimating the return in ferrocyanide of the melted mass by the old process. There are also several methods known for the estimation of the Prussian blue in old purifying material (C. Moldenhauer and W. Leybold, *Journ. f. Gasbel.*, 1889, 155; Knublauch, *ibid.*, 1889, 450; Gasch., *ibid.*, 1889, 166; Drehschmidt, *ibid.*, 1892, 221).

Frequent analyses of old purifying material have made us endeavour to find a more rapid method, which would at the same time be as exact, for the estimation of ferrocyanides besides those already published.

The method we have devised rests on the following facts:—The alkaline ferrocyanides, as well as the ferricyanides, in acid solution, energetically resist the action of oxidising agents even when these are fairly strong, while they are easily decomposed by the same reagents in alkaline solution. Under these conditions all the iron is separated in the form of a precipitate composed principally of ferric oxide, and is easy to estimate quantitatively.

When we place in contact a solution of potassic ferrocyanide and bromised soda, at the ordinary temperature (80 grms. of sodic hydrate is dissolved in water, and after cooling made up to 1 litre, 20 c.c. of bromine are then added while stirring vigorously) no reaction takes place; but, after prolonged heating, a thick pulverulent heavy precipitate, of a brick-red colour, is formed, and at the same time there is a lively disengagement of gas.

Qualitative analyses have shown that in this reaction the ferrocyanide is first transformed into ferricyanide, and this latter, being submitted in its turn to oxidation, is decomposed in the manner just described. The reaction therefore has two phases. On leaving the precipitate

formed to stand for some hours, then filtering and washing it, the iron can be estimated by any well-known method, and the proportion of ferrocyanide present calculated. It suffices to multiply the weight of the iron found by 7·5476 to get the amount of the crystallised salt $K_2FeCy_6+3H_2O$, or by 6·5833 for the anhydrous salt.

The iron in this precipitate may be estimated by several different methods.

The precipitate can be dissolved on the filter by means of dilute warm hydrochloric acid, and the iron precipitated in the hydrochloric solution by ammonia. We can also dry the precipitate on the filter; put as much of the precipitate as possible into a small flask, incinerate the filter, decompose the small quantity of ferric oxide with bisulphate of potassium, add it to the rest of the precipitate, and dissolve the whole in dilute sulphuric acid. It must then be reduced by zinc and titrated with permanganate.

The analyses made by us gave very concordant results. Supposing the product obtained contained 99·6 per cent of crystallised potassic ferrocyanide, the results we obtained by this process were from 0·2 to 0·4 per cent too low.

The bromised soda as an oxidising agent can be replaced by permanganate of potash in alkaline solution. When we submit a solution of potassic ferrocyanide and an alkaline solution of permanganate of potash to the action of heat, the latter body is rapidly reduced under these conditions; there is evidently formed a precipitate of hydrated peroxide of manganese, besides the precipitate referred to above, and containing a quantity of ferric oxide corresponding to the potassic ferrocyanide present. By continuing the addition of the permanganate until the red tint remains constant, and adding dilute hydrochloric acid and heating, this precipitate, as well as the excess of permanganate, is decomposed and chlorine given off. It suffices to drive off the chlorine by heat, to separate the iron from the manganese by the acetate method, in the sufficiently diluted solution, to wash and calcine the precipitate of manganiferous basic ferric acetate, and to estimate volumetrically by permanganate the quantity of iron present.

In every case the use of bromised soda is preferable, in spite of its higher price, as its action is much more rapid.

To apply the process just described to old purifying material, the following is the best manner of proceeding:—Old purifying material contains, besides ferrocyanides, sulphur and a considerable amount of non-volatile tarry products. To estimate the Prussian blue, or rather the ferrocyanides, the material should be finely powdered, and treated at a gentle heat with an excess of caustic potash at about 15 per cent, with a view to completely transforming the Prussian blue into ferric hydrate and ferrocyanide of potassium. After dilution, the alkaline liquid obtained is filtered and the residue washed, after having been treated on the filter with a small quantity of dilute potash lye, until the whole of the ferrocyanide has disappeared.

However, we eventually discontinued the use of this somewhat complicated and tedious method of decomposition, and adopted the method given by Cr. Moldenhauer and W. Leybold (*Journ. f. Gasbel.*, 1889, p. 157).

The spent material is rapidly ground up in an iron mortar, well mixed, and 50 grms. is taken for the analysis. This sample is placed in a flask of 1 litre capacity, and moistened with 100 to 150 c.c. of caustic potash at 15 per cent. The flask is then left for some time in a warm place, and frequently shaken; it is then filled up to 1030 c.c., as the results obtained by the above-mentioned chemists show that the residue of 50 grms. of material occupies a volume of 30 c.c. The liquid is transferred to a larger flask, well mixed, and filtered through a folded filter-paper. The potassic ferrocyanide is estimated in an aliquot part of the filtrate, the colour of which varies from brownish-yellow to reddish-brown, by the method already described. We recommend, by preference, the volumetric estimation of the iron in sulphuric solution by permanganate, after reduction to the ferrous state.

More exact results can be obtained by exhausting the material by means of sulphide of carbon, with the object of eliminating the sulphur and the greater part of the tarry products, previous to proceeding in the manner just described.

The analysis, by our method, of a particularly rich sample of an old purifying material, gave us the following results, calculated as crystallised ferrocyanide of potassium, 14·3, 14·54, 14·51, and 14·65 per cent; another method gave 14·48 per cent.

Although we have not had the opportunity of applying our method of analysis to the estimation of the ferrocyanides in the melted mass of the old process, we have no doubt but that, in this case also, this method would give entirely satisfactory results.

We may further mention that our method is not confined to the estimation of the ferrocyanide corresponding to Prussian blue, but it may also be used in connection with the ferrocyanides corresponding to the other hydroferrocyanic compounds which may be met with in all purifying material.—*Zeitschrift für Angewandte Chemie*, 1899, No. 15, p. 345.

THE TITRATION OF PERSULPHATES.

By MM. LEBLANC and ECKARDT.

The discovery made by Marshall (*Journ. Chem. Soc.*, lix., 771; *Mon. Scient.*, Nov., 1897, p. 818) of a practicable method for the preparation of persulphates, has given rise to a considerable amount of research on this class of bodies. According to Marshall, Elbs (*Journ. f. Prakt. Chem.*, xlviii., 185; *Zeit. Elektr.*, i., 417; ii., 245; *Zeit. Ang. Chem.*, 1897, 195) and Schönherr were occupied with the electrolytic production of the persulphates and the study of their reactions. With regard to their physical properties these bodies were studied by Berthelot (*Comptes Rendus*, cxiv., 875), Löwenhertz (*Chem. Zeit.*, xvi., 838), Bredig (*Zeit. Phys. Chim.*, xii., 230), and Möller (*Zeit. Phys. Chim.*, xii., 555).

Persulphuric acid and its salts have already been used industrially, and are now beginning to be used in considerable quantities. It is, however, difficult to say at present whether the use of persulphate will be further developed, since the scientific study of these bodies is not yet complete.

However this may be, it is quite understood that at the present time it is a matter of great interest to find a simple method for the rapid estimation of the amount of active oxygen in a solution of a persulphate. With this object Marshall (*loc. cit.*), Löwenhertz (*loc. cit.*), Möller (*loc. cit.*), Elbs (*loc. cit.*) and Schönherr have already proposed adding to the persulphate an excess of a solution of a ferrous salt, and to titrate the excess with permanganate. None of these writers, however, have given any indication as to the method of working to be followed; they have all limited themselves to showing the accuracy of the method by check analyses made in a different manner. Poleck's remark (*Chem. Zeit.*, xx., 631), according to which it is necessary to add to 0·2 grm. of persulphate 4 to 4½ times its weight of the iron salt, appears to be somewhat strange.

Being occupied in the manufacture of persulphates and in measuring their powers of oxidation, we have had recourse to the methods in question, and we immediately met with certain difficulties. The salts we prepared showed nothing but an absolutely insignificant power of oxidation, not only in our own hands, but also in those of other chemists who undertook their analysis. Further, the results of the analyses were far from being concordant. We therefore decided to study the question much more closely, and we almost immediately discovered the cause of the anomaly. The reaction between the persulphate and

the salt of protoxide of iron only takes place very slowly at the ordinary temperature of the laboratory, which will explain the low results obtained. The error, in fact, may reach 20 or even 30 per cent. On the other hand, at high temperatures, we can easily obtain very exact results.

We will give a few examples which will quite clear up the question:—The salt decomposes according to the equation, $K_2S_2O_8 = K_2SO_4 + SO_2 + O_2$.

By the loss of weight, therefore, we can determine the value in persulphate of the sample analysed. Our results were as follows:—

1. 0·5995 grm. $K_2S_2O_8$ gave 0·3845 grm. of residue.
2. 0·6030 „ „ „ 0·3865 „ „

If we admit, what is extremely probable, that the residue is formed entirely of sulphate of potassium, and that this sulphate exists in the state of persulphate in the original sample, it is easy to deduce that the proportion of persulphate in the salt analysed was 99·54 per cent according to the first analysis, and 99·50 per cent according to the second. It is evident that this salt was contaminated with a small quantity of persulphate of ammonia.*

We dissolved 2·474 grms. of persulphate, and made the volume of the liquid up to 100 c.c. For each titration we took 10 c.c. of this solution, to which we added 5 c.c. of sulphuric acid of 1·16 density. We then added an excess (1 to 10 c.c. too much) of a solution of the double sulphate of iron and ammonia (at 30 grms. per litre), and titrated the excess of iron by means of a solution of permanganate of potash at 2·4 grms. per litre.

Below we give a description of the different experiments made.

1. To the mixture prepared in the manner we have just described, we added about 100 c.c. of distilled water at 70—80°, then, without further heating, titrated with permanganate. The permanganate itself had already been titrated in duplicate by means of pure Mohr's salt, and with oxalic acid. The following were the results:—

(*a*) 24·67, (*b*) 24·60 c.c. of solution, which gave, for the solid salt analysed, (*a*) 99·71 per cent, (*b*) 99·79 per cent of pure persulphate of potash.

2. The preceding mixture was prepared in an atmosphere of carbonic acid; 100 c.c. of boiled distilled water are added, the whole heated to 60° and kept at that temperatue for ten minutes. It was then titrated with permanganate. The results were as follows:—

(*a*) 24·66, (*b*) 24·65 of iron solution, which corresponds to (*a*) 99·68 per cent, (*b*) 99·63 per cent, of pure persulphate of potash in the original sample.

As can be seen from the first experiment, we obtain good results by raising the temperature, without driving off the air. The same result could not be obtained at the ordinary temperature except after a much longer time.

3. The same mixture was diluted with 100 c.c. of water and left in contact with carbonic acid at the ordinary temperature for half an hour. (Air produced exactly the same effect). The solution was then titrated with permanganate, and the following results obtained:—

(*a*) 24·62, (*b*) 24·60 c.c. of iron solution, which corresponds to (*a*) 99·51, (*b*) 99·68 per cent of pure persulphate of potash in the sample. On the other hand, by not taking any precautions, and operating in the presence of a variable excess of the ferrous solution, we obtained the following results:—

4. One c.c. of iron solution in excess, and titrating with permanganate *as rapidly as possible*. Result: 14·41 c.c. of iron solution = 58·25 per cent of persulphate.

5. Four c.c. of iron solution in excess, titrate rapidly. Result: 18·96 c.c. of iron solution = 76·66 per cent of persulphate.

6. Five c.c. of iron solution in excess, titrate rapidly. Result: 20·22 c.c. of iron solution = 81·74 per cent of persulphate.

7. Ten c.c. of iron solution in excess, titrate rapidly. Result: 22·32 c.c. of iron solution = 90·22 per cent of persulphate.

8. Twenty c.c. of iron solution in excess, titrate rapidly. Result: 23·60 c.c. of iron solution = 95·39 per cent of persulphate.

9. One hundred c.c. of iron solution in excess, titrate rapidly. Result: 24·64 c.c. of iron solution = 99·59 per cent of persulphate.

10. One hundred c.c. of iron solution in excess, titrate rapidly. Result: 24·60 c.c. of iron solution = 99·44 per cent of persulphate.

To sum up, the reaction is the more rapid the greater the excess of iron salt present, a result that could be foreseen according to the law of masses. It is also probable that the ferrous salt exerts a catalytic action. Poleck's observation is therefore not unreasonable; it is, however, much simpler to titrate warm, and thus avoid the large excess of the iron salt.

We have further endeavoured to find whether, instead of working at a high temperature, we could not make use of a catalytic material; but up to the present we have not obtained any favourable result. There is nothing to be expected from metallic salts, since the solution already contains iron and manganese in large quantities. Platinised asbestos, such as is used in the manufacture of sulphuric anhydride, has not given any result.—*Zeitschrift für Elektrochemie*, 1899, p. 355.

* This method of estimation does not enable us to form any conclusion as to the oxidising power of the substance. If, for example, we mix 174 grms. of sulphate of potassium and 96 grms. of sulphate of ammonia, and submit this mixture to the examination in question, we could deduce a proportion of persulphate of potash present of 100 per cent, while the true quantity would be 0 per cent.

CONTRIBUTION TO THE ASSAY OF GOLD.

By W. WITTER.

Not long ago Bock (*Chemiker Zeitung*, 1897, p. 973, and 1898, p. 358), published a new method for the assay of gold, a method which had none of the disadvantages of the old process, while being quite as rapid. I have made a large number of experiments with a view to proving the accuracy of the old method, and to examine the advantages offered by Bock's new method. I proposed to decide the following questions:—1. The difference which exists between the commercial assay and scientific analysis; 2. Is cupellation, in our present state of knowledge, sufficiently exact to serve as a basis for commercial transactions? 3. Is an analysis of any practical use, from the point of view of loss of interest, in the case of large quantities of gold? 4. What advantages has Bock's method over the usual method? 5. How far can it be trusted?

In cupellation, temperature plays an important part.

According to Rose ("Metallurgy of Gold," 1896, p. 441), there is a loss of 0·01 per thousand of gold for a rise of temperature of 5°. I have succeeded, with the help of a platinum-rhodium couple, and a Keiser-Schmidt galvanometer, in maintaining a constant temperature during the whole operation, and thus obtained results conforming to those given by wet analysis. For chemically pure gold, it is necessary to have a temperature of 960°, less fine gold requires 950—960°, and gold containing small quantities of platinum requires a temperature of 1000—1010°. Ductile gold, 973 fine, containing both silver and copper, requires practically the same temperature as chemically pure gold. Gold coin, containing 100 parts of copper to 900 parts of pure gold, should be heated to only 920—930°, while for gold only 720 fine, containing silver and copper, a temperature of 900° is even too high.

When a good pyrometer is not obtainable, alloys melting at temperatures varying by 20—25° from 850—1050 can be used.

The Frankfurter Scheideanstalt prepares these alloys. By this means very satisfactory results can be obtained, not differing more than 0·02 per cent or 0·2 per thousand,

With regard to questions 2 and 3, the variations noticed, 0·0—0·2 per thousand, are extremely small, and show that cupellation can well be used as a basis for commercial transactions.

The results obtained by Bock's method are a little higher, say 0·2 per thousand. Although the old method is sufficiently exact, Bock's process can, in certain cases, be employed with great advantage, above all when it is necessary to make a number of analyses of one and the same substance. This method requires less manipulative skill on the part of the operator than does cupellation, and temperature has no influence at all. Unfortunately it is up to the present only applicable to the assay of gold coin.—*Chemiker Zeitung*, 1899, p. 522.

THE ESTIMATION OF SULPHYDRIC, SULPHUROUS, AND HYPOSULPHUROUS ACIDS.

By W. FELD.

THE methods described in this paper are applicable to the alkaline or alkaline-earthy salts of the above mentioned acids, even when they are present in extremely small quantities.

1. *Sulphides.*—The alkaline or alkaline-earthy sulphides give up the whole of their sulphur as sulphuretted hydrogen when boiled with a concentrated solution of chloride of magnesium in an atmosphere of carbonic acid.

The powdered sample, moistened with water, is placed in a conical flask of 300 c.c. capacity, fitted with an india-rubber stopper with two holes. Through one hole passes the stem of a funnel, fitted with a tap, and reaching to the bottom of the flask; through the other hole passes the escape tube which leads the gas through four wash-bottles. The fourth bottle is connected to a 10-litre flask serving as an aspirator. The opening of the funnel is fitted with a cork carrying a tube connected to a source of carbonic acid. This carbonic acid should not have any action on a solution of iodine. The first wash-bottle is empty, the second and third contain a quantity of a solution of iodine, more than sufficient to absorb the whole of the sulphuretted hydrogen disengaged; and finally, the fourth wash-bottle contains a decinormal solution of hyposulphite, for the purpose of absorbing the traces of iodine which might be carried through the preceding bottles by the current of carbonic acid gas.

We begin by passing through about 1 litre of carbonic acid for the purpose of driving the air out of the apparatus. The top of the funnel tube is then closed, and the stopper taken out of the funnel neck; 20 c.c. of a 25 per cent solution of chloride of magnesium are then poured in, and the rubber stopper replaced, and, by opening the tap, the chloride of magnesium is run into the flask, which already contains the sample. Heat slowly to boiling, while continuously passing carbonic acid gas through at the rate of 10 litres in three-quarters of an hour. The operation is generally finished at the fifth litre. The wash-bottles are then disconnected, emptied, and carefully washed out with distilled water; the whole of the liquids are then added together and titrated as one.

The reactions are as follows:—

1. $BaS + MgCl_2 + CO_2 + H_2O = BaCl_2 + MgCO_3 + H_2S$;
2. $H_2S + 2I = 2HI + S$.

2. *Sulphites.*—Proceed as for sulphides, but replacing the chloride of magnesium by hydrochloric acid.

3. *Hyposulphites.*—Hyposulphites give off a little sulphuretted hydrogen when treated with hydrochloric acid; the following is a method which gives exact results:—

The hyposulphite is first transformed into tetrathionate by titration with a solution of iodine. The solution of tetrathionate, diluted with 50 c.c. of water, is placed in a flask with an excess of aluminium foil, and treated with *dilute* hydrochloric acid, in an atmosphere of carbonic acid, in the cold. The reduction to sulphuretted hydrogen is effected quantitatively according to the equation—

$$Na_2S_4O_6 + 20HCl + 6Al = 2NaCl + 3Al_2Cl_6 + 6H_2O + 4H_2S.$$

4. *Hyposulphite in the presence of Sulphite.*—This estimation is made by method 3; the titration with iodine oxidises the sulphite into sulphate, which is not modified by nascent hydrogen.

5. *Sulphite in the presence of Hyposulphite.*—Add to the substance an excess of bichloride of mercury; the hyposulphite is thus transformed into mercuric sulphide according to the equation—

$$Na_2S_2O_3 + HgCl_2 + H_2O = Na_2SO_4 + HgS + 2HCl.$$

The sulphate is not modified by this reaction, and can be determined by method 2.

6. *Sulphide, Sulphite, and Hyposulphite.*—The sample is first treated with chloride of magnesium, as described in par. 1. In this manner the sulphide is estimated. The wash-bottles are refilled, and an excess of bichloride of mercury is then added to the contents of the flask, in the cold; then distil with hydrochloric acid, as described in par. 2. We thus obtain the sulphite. Finally, the hyposulphite is determined on a fresh portion of the sample, by titrating with iodine; the sulphide is thus transformed into sulphur, and the sulphite into sulphate. There now remains only the reduction by nascent hydrogen as described in par. 3.

If, besides the alkaline and alkaline-earthy salts enumerated above, the substance contains polysulphides, free sulphur, or sulphides of the heavy metals, the difficulties of titration are much greater, and have not yet been satisfactorily overcome. In the meantime, however, fairly good results have been obtained by the following method of procedure:—

The free sulphur is extracted with bisulphide of carbon and weighed after evaporating off the solvent. The sulphur present in the form of sulphide is then estimated by the method given in par. 1. In this operation the sulphur of the polysulphides is given off partly in the state of sulphuretted hydrogen, the remainder separating in the free state.

This latter portion is extracted with bisulphide of carbon. In any case, if the sample contains a sulphite, a little hyposulphite is formed. The solution is then titrated with iodine. During this operation the sulphur present in the state of ferrous sulphide separates in the free state, and can be extracted with bisulphide of carbon. The solution is then treated by method 3, to estimate the hyposulphites. Here the presence of other polythionic acids might introduce an error.

In solid substances, sulphites may occur in the presence of polysulphides; in such a case they are estimated by treatment with bichloride of mercury and distillation with hydrochloric acid, according to method 2.—*Die Chemische Industrie*, xxi., p. 372—380.

Estimation of Boric Acid in Boracite.—Dr. Rudolph Schwartz.—The disaggregation of boracite is effected in the following manner: 1 to 2 grms. of the finely powdered material are treated with 5 to 10 c.c. of hydrochloric acid at 1·124, and 100 c.c. of water, and heated for about twenty or thirty minutes on the water-bath in a flask fitted with a vertical condenser, and then left to stand for several hours. It is then filtered, washed thoroughly, and neutralised exactly by means of a N/5 solution of soda, in the presence of two or three drops of methyl orange. The solution thus obtained is poured into a 100 or 200 c.c. flask, and made up to the gauge mark with water; 50 c.c., equal to 0·2 or 0·4 grm. of material, are taken, this is diluted to 150—200 c.c., and titrated with an N/5 solution of soda in the presence of phenolphthalein. To more easily note the exact termination of the titration, that is the production of the red colouration, it is advisable to add 50—60 per cent of glycerin; this latter must of course be perfectly neutral, and must be carefully neutralised if necessary.—*Chemiker Zeitung*, 1899, p. 497.

ON THE

VOLUMETRIC ESTIMATION OF CERIUM.*

By PHILIP E. BROWNING.

(Concluded from p. 31).

The Action of Arsenious Oxide upon Cerium Dioxide (with WM. D. CUTTER).

THE fact that cerium dioxide is reduced by hydriodic acid suggested the possibility of the application of arsenious acid in acid solution to the same end, according to the reaction $4CeO_2+As_2O_3=2Ce_2O_3+As_2O_5$.

The extreme difficulty with which the ignited cerium dioxide when pure dissolves in acids has already been mentioned, and for this reason it was found practically impossible to obtain any results by this method. Weighed portions of the dioxide were placed in Erlenmeyer beakers with an excess of a solution of arsenious oxide $\frac{1}{10}$ N, 10 c.m.³ of (1 : 1) sulphuric acid were added, and the boiling continued until the fuming-point of the acid was reached; but even at this point only a partial solution of the dioxide had taken place.

The dark brown powder obtained by igniting the carefully washed oxalates, precipitated in acid solution by treating a solution of crude cerium chloride with ammonium oxalate or oxalic acid, is very fairly soluble in acids. Mengel (*Zeit. Anorg. Chem.*, xix., 67) has recently shown that this product contains a dioxide of praseodidymium which acts as does cerium dioxide toward reducing agents. This fact makes the results recorded in the treatment of this ignited mixture of oxides of no value analytically, but of interest in the comparative study of the two reducing agents—arsenious oxide and hydriodic acid. Two portions of this mixture of oxides gave the following results, which agree fairly well with those of Mengel.

	Amount of substance taken.	CeO_2+(PdO_2?) found.	Calculated on 0·1000 grm.
	Grm.	Grm.	Grm.
1.	0·1037	0·0530	0·0511
2.	0·1034	0·0538	0·0520

The average of these results was taken as a standard —0·0515 grm. CeO_2, &c., to every 0·1000 grm. of material. Three carefully weighed portions of this same material were placed in Erlenmeyer beakers with 10 c.m.³ of $\frac{1}{10}$ N arsenious oxide solution and 10 c.m.³ of dilute (1 : 4) sulphuric acid, and boiled until complete solution had taken place. The liquid was then cooled, neutralised with potassium bicarbonate, and titrated with standardising iodine to determine the amount of arsenious oxide remaining, and from it the amount used in the reduction of the dioxide according to the reaction given above. The results obtained follow.

	Amount taken.	Amount CeO_2 found.	CeO_2 calculated for 0·1000 grm.
	Grm.	Grm.	Grm.
1.	0·1005	0·0493	0·0491
2.	0·1015	0·0494	0·0487
3.	0·1005	0·0486	0·0484

As will be seen, the results obtained by this method fall about 0·0030 grm. below the standard as obtained by the distillation method, which seems to show that the arsenious oxide does not effect the complete reduction of the cerium dioxide from CeO_2 to Ce_2O_3.

In order to study this point a little more fully and upon the pure dioxide, definite portions of a standard solution of pure cerium chloride were precipitated by ammonia in the presence of hydrogen dioxide and boiled to reduce the CeO_3 formed to the conditions of CeO_2. The precipitated hydrated dioxide was filtered off and carefully washed until the washings gave no indication of hydrogen dioxide. The moist precipitate was then washed into a beaker, 1 grm. of potassium iodide added, and 10 c.m.³ of strong HCl. The precipitate dissolved quite readily in the cold, and the iodine liberated was determined by standard sodium thiosulphate. The results appear in Table V.

TABLE V.

	CeO_2 taken.	CeO_2 found.	Error.
	Grm.	Grm.	Grm.
1.	0·1142	0·1140	0·0002 —
2.	0·1142	0·1147	0·0005 +
3.	0·1142	0·1152	0·0010 +
4.	0·1142	0·1159	0·0017 +
5.	0·1142	0·1152	0·0010 +
6.	0·1142	0·1156	0·0014 +

Another series of these precipitates prepared in the same way was boiled with a definite amount of arsenious acid in acid solution, as previously described in the case of the ignited dioxide. The results, which are recorded in Table VI., show, as in the case of the ignited dioxide, an insufficient reduction of the cerium by the arsenious acid.

TABLE VI.

	CeO_2 taken.	CeO_2 found.	Error.
	Grm.	Grm.	Grm.
1.	0·0381	0·0370	0·0011 —
2.	0·0381	0·0361	0·0020 —
3.	0·1142	0·1077	0·0064 —
4.	0·1060	0·1002	0·0058 —

The Estimation of Cerium Oxalate by Potassium Permanganate (with LEO. A. LYNCH).

Stolba (*Sitzungsber. d. kgl. böhm. Gesellsch. d. Wissenschaften*, iv., July, 1879; *Zeit. Anal. Chem.*, xix., 194) has stated that cerium oxalate may be estimated volumetrically after the same manner as calcium oxalate by treating the washed precipitate, suspended in warm water, to which a moderate amount of sulphuric acid has been added, by potassium permanganate. As the titration proceeds the precipitate disappears and the end-reaction is sharp. He also finds that the permanganate does not oxidise the cerium from the lower to the higher condition. So far as we have been able to discover, no experimental evidence has been presented to prove the correctness of Stolba's statement, and the work to be described was undertaken to furnish such evidence.

The solutions used were prepared and standardised as follows:—The cerium solutions were made by dissolving 10 grms. of pure cerium chloride in 1 litre of water, and standardised by precipitating measured and weighed portions in a faintly acid solution, with ammonium oxalate, filtering, washing, igniting, and weighing as the dioxide (CeO_2). A solution of potassium permanganate was prepared and standardised by titration against weighed amounts of ammonium oxalate. A solution of ammonium oxalate was made and its value determined by titrating measured amounts against potassium permanganate. Definite portions of the cerium solution were drawn from a burette and after diluting with water from 100 to 200 c.m.³ a definite amount of ammonium oxalate was added, care being taken to have an excess over the amount necessary, and the whole warmed to ensure a more crystalline precipitation. (As shown by the table, the precipitation was sometimes in neutral, sometimes in faintly acid solution). The precipitate was then filtered off on paper and carefully washed, the filtrate and washings being collected in a litre Erlenmeyer flask and set aside for future use. The precipitate was treated with about 10 c.m.³ of hot (1 : 4) sulphuric acid, which dissolved it completely, if not at first, by running it through the filter a few times, and the solution and washings were collected in another litre flask. The total volume of liquid was made up to about 500 c.m.³, warmed to about 70—80° C., and titrated with potassium permanganate to the appearance of the faint blush of colour showing the complete

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, 1899, vol. viii., No. 48.

oxidation of the oxalic acid. The filtrate from the cerium oxalate containing the excess of oxalic acid was diluted to 500 c.m.^{3}, acidified with 10 c.m.^{3} of dilute (1 : 4) sulphuric acid, 1 grm. of manganous sulphate added to prevent the interfering action of the free hydrochloric acid upon the estimation of the oxalic acid (Gooch and Peters, *Am. Journ. Sci.*, vii., 461), and titrated with potassium permanganate after the same manner as the dissolved precipitate. A definite quantity of ammonium oxalate having been originally taken, it became possible, by subtracting from it the amount obtained, to derive the measure of the oxalate used in the precipitation of the cerium oxalate. By this procedure, it will be observed a check was made upon the results obtained by the titration of the precipitate. In Experiments 1 to 6 the cerium oxalate was thrown down in neutral solution, in Experiments 7 to 10 in acid solutions. The treatment of the filtrate in Experiment 1 was made without the presence of the manganous sulphate. The results recorded in Table VII. seem to uphold the statement of Stolba.

TABLE VII.

	Amount taken. Calculated as $CeCl_3$.	Amount found. Calculated as $CeCl_3$. Treatment of precipitate.	Error. Calculated as $CeCl_3$.	Amount found. Calculated as $CeCl_3$. Treatment of filtrate.	Error. Calculated as $CeCl_3$.
1.	[0·1091	0·1087	0·0004 −	0·1023	0·0068 −]
2.	0·1091	0·1103	0·0012 +	—	—
3.	0·1091	0·1087	0·0004 −	0·1087	0·0004 −
4.	0·1364	0·1373	0·0009 +	0·1391	0·0027 +
5.	0·1364	0·1367	0·0003 +	0·1367	0·0003 +
6.	0·2182	0·2202	0·0020 +	0·2206	0·0024 +
7.	0·1091	—	—	0·1087	0·0004 −
8.	0·1519	0·1535	0·0016 +	0·1535	0·0016 +
9.	0·1364	0·1367	0·0003 +	0·1367	0·0003 +
10.	0·2182	0·2183	0·0001 +	0·2183	0·0000

A CONTRIBUTION TO THE STUDY OF LIQUID MIXTURES OF CONSTANT BOILING-POINT.*

By GARNETT RYLAND.

(Continued from p. 16).

Methyl Alcohol and Benzene.

Method of Analysis.—The distillates were analysed by the following method:—

Thirty to forty c.c. of the fraction boiling at a constant temperature were measured off into a burette and a definite proportion of water added. It was found that when alcohol constituted 30—40 per cent of the distillate the water added should be one-third of the volume of the sample. The burette was closed, shaken vigorously, and allowed to stand until the liquid within separated into two sharply divided layers of benzene and aqueous alcoholic solution. The volume of benzene could then be read off and its volume ratio to the alcohol converted into the weight ratio by determining the specific gravity of each liquid.

(The difference between the actual specific gravity of a solution of benzene in methyl or ethyl alcohol and that calculated from the specific gravities of the individual liquids was found by Paterno and Montemartini (*Gazz. Chim. Ital.*, 1894, xxv., II., p. 179) to be so small (1 or 2 in the third decimal place) that it may be disregarded in this method of determination).

A series of six test determinations of known mixtures gave the following results for the per cent of benzene by volume:—

	Per cent of benzene.					
	I.	II.	III.	IV.	V.	VI.
Calculated ..	58·8	59·5	59·5	60·0	64·0	64·0
Found	58·0	59·4	59·8	60·5	63·5	64·3

Results.— Methyl alcohol (distilled from lime at 64·5—65°)* and commercial benzene (re-distilled at 79—79·5°) were mixed in the proportions by weight, and distilled at the pressures given below. The constant boiling portions analysed were separated from the excess of either constituent by three fractional distillations in each case (see Table A).

Ethyl Alcohol and Benzene.

Mixtures of ethyl alcohol (distilled from anhydrous copper sulphate at 77·5—78°) and benzene (79—79·5°) were investigated, and it was found that a mixture containing about 32 per cent of alcohol was distilled to the last drop at 67—68°.

The results of the analysis of distillates under various pressures, obtained as in the case of methyl alcohol and benzene, are given in Table B.

To determine the percentage composition of the distillates, 40 c.c. were shaken with 50—60 c.c. of water.

Twelve analyses of known mixtures gave the following results for the per cent of benzene by volume:—

* From the *American Chemical Journal*, vol. xxiii., No. 5, Nov., 1899.

* The same thermometer was used throughout this and all succeeding investigations. It had been carefully tested, compared with two standard thermometers, and corrected. Whenever the pressure under which a liquid was distilled is not stated, it was approximately 760 m.m.

TABLE A.

	Ratio of alcohol to benzene (by weight) in mixture distilled.	Pressure. M.m.	Boiling-point of fraction analysed.	Per cent of alcohol found.	Per cent of benzene found.	Ratio of products of vapour-tension by vapour density.*
I.	38 : 62	770	58°	38·4	61·6	At 60°, 37·8 : 62·2
II.	38 : 62	760	57·5—58°	39·0	61·0	
III.	37 : 63	400	40°	36·8	63·2	At 40°, 35·2 : 64·8
IV.	36 : 64	392	39·5°	35·7	64·3	
V.	41 : 59	223	25°	33·2	66·8	At 25°, 33·2 : 66·8
VI.	34 : 66	223	25°	33·1	66·9	

* See Berthelot and Thorpe, p. 16 (*ante*). The values for vapour-tensions throughout these investigations were taken from Landolt and Börnstein's tables, Regnault's values being preferred.

TABLE B.

	Ratio of alcohol to benzene (by weight) in mixture distilled.	Pressure. M.m.	Boiling-point of fraction analysed.	Per cent of alcohol found.	Per cent of benzene found.	V. t. × v. d. of alcohol / V. t. × v. d. of benzene.
I.	33 : 67	769	67—68°	31·4	68·6	At 67·5°, 36·3 : 63·7
II.	33 : 67	763	67—68°	32·3	67·7	
III.	33 : 67	421	50—51°	28·2	71·8	At 50°, 32·3 : 67·7
IV.	28 : 72	423	50—51°	27·6	72·4	
V.	28 : 72	241	34·5—35·5°	23·3	76·7	At 35° 28·9 : 78·1
VI.	23 : 77	243	34·5—35·5°	23·4	76·6	

	I.	II.	III.	IV.	V.	VI.
Calculated ..	60·0	60·0	60·0	65·0	65·0	65·0
Found	59·9	59·9	62·2	65·2	64·8	65·0
	VII.	VIII.	IX.	X.	XI.	XII.
Calculated ..	65·0	70·0	70·0	70·0	75·0	75·0
Found	66·2	70·1	69·8	70·0	75·0	75·1

(To be continued).

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING DECEMBER 31ST, 1899.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, January 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Dec. 1st to Dec. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month was 1·30 inches. The average for the past thirty years is 2·10 inches; this leaves a deficiency of 0·80 inch, bringing the total deficiency for the year up to 4·54 inches, or 17·6 per cent, as will be seen by the following table:—

Rainfall in Inches at Oxford, Month by Month, during the Year 1899.

	Actual fall.	Mean of 30 years.	Difference from the mean.	
	Inches.	Inches.	Inch.	Inch.
January ..	2·85	2·16	—	+0·69
February ..	1·92	1·76	—	+0·16
March ..	0·25	1·50	−1·25	—
April	1·83	1·66	—	+0·17
May	1·37	1·83	−0·46	—
June	1·05	2·11	−1·06	—
July	1·32	2·68	−1·36	—
August ..	1·80	2·32	−0·52	—
September..	2·38	2·43	−0·05	—
October ..	2·59	2·75	−0·16	—
November ..	2·52	2·42	—	+0·10
December ..	1·30	2·10	−0·80	—
	21·18	25·72	−5·66	+1·12

During the month of December the bacteriological examinations of 502 samples gave the results recorded in the following table; we have also examined 34 other

Table showing the Average Monthly Bacterial Variations during the Year.

Month.	Thames, unfiltered.	Thames-derived Companies, filtered.	New River, unfiltered.	New River, filtered.	River Lea, unfiltered.	River Lea (East London), filtered.	
January	22764	35	1651	11	4576	14	
February	23280	54	2995	19	7481	68	
March	12133	28	2032	35	3977	33	
April	10622	22	908	8	2490	5	
May	3247	26	430	15	1313	11	
June	1677	30	408	20	1225	19	
	12287	32	1404	18	3510	25	Mean of 1st 6 months.
July	1270	41	1858	30	4357	26	
August	2269	31	410	12	863	12	
September	574	16	170	4	220	9	
October	1367	20	290	10	876	21	
November	3724	11	355	13	513	7	
December	1883	14	387	4	590	10	
	1848	22	578	12	1236	14	Mean of 2nd 6 months.
	7067	27	991	15	2373	20	Mean of 12 months.

Averages of the Chief Chemical Composition of the Six Supplies derived from the River Thames.

	Common salt per gallon.	Nitric acid per gallon.	Hardness. Degrees.	Oxygen required per gallon.	Organic carbon per gallon.	Colour. Brown : Blue.
January	2·236	1·032	15·73	0·072	0·227	25·4 : 20
February	2·144	1·107	15·75	0·073	0·207	26·6 : 20
March	2·092	1·014	16·14	0·037	0·134	12·5 : 20
April	2·147	0·932	14·43	0·031	0·093	8·8 : 20
May	2·235	0·868	13·67	0·036	0·102	11·5 : 20
June	2·150	0·724	13·40	0·033	0·095	10·5 : 20
July	2·150	0·623	13·04	0·035	0·085	10·2 : 20
August	2·202	0·756	13·00	0·035	0·086	13·8 : 20
September	2·174	0·677	13·08	0·032	0·083	14·5 : 20
October	2·198	0·638	13·85	0·028	0·084	11·9 : 20
November	2·439	0·771	14·85	0·058	0·155	24·0 : 20
December	2·432	0·884	17·01	0·036	0·132	19·9 : 20

samples, from special standpipes, &c., making a total of 536 samples :—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	387
New River, filtered (mean of 124 samples) ..	4
Thames, unfiltered (mean of 24 samples) ..	1883
Thames waters, from the clear-water wells of eight Thames-derived supplies (mean of 270 samples)	14
Ditto ditto .. .. highest	154
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 24 samples) ..	590
River Lea, from the East London Company's clear-water well (mean of 36 samples) . ..	10

The chief remark to be made with regard to the supply during December is to draw attention to the exceptional bacterial purity of the unfiltered waters of the Thames, New River, and the Lea. The general supply has been admirable from a bacteriological point of view.

During the past year we have examined 4792 samples of London water bacteriologically, as compared with 3500 and 3249 respectively in the two previous years. We have also made 2456 chemical analyses of London waters throughout this period, making a total of 7248 samples examined.

The accompanying table shows that during the first six months the Thames-derived Companies' clear water wells contained on the average 32 bacteria per c.c., while the New River and River Lea supplies contained respectively 18 and 25. During the second six months the number of bacteria in the waters from the three were respectively 22, 12, and 14. These results show that during the year effective filtration of the London waters has been properly maintained.

When we consider that a water containing about 100 bacteria per c.c. in the clear water wells would be regarded by the highest authorities as properly filtered, we see that the London supply must be considered exceptionally good.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

NOTICES OF BOOKS.

Inorganic Chemistry for Advanced Students. By Sir Henry Roscoe, F.R.S., D.C.L., LL.D., and Arthur Harden, Ph.D., M.Sc. (Vict.). With Fifty-four Illustrations. London: Macmillan and Co., Ltd. 1899. Pp. 432.

The present work is intended as a Supplement to the smaller volume of "Inorganic Chemistry for Beginners," published six years ago, and is therefore for the use of students who have already had some practical experience in the Science. A useful feature of this book, which is not often met with in books for students, is a number of lessons on the application of chemistry to the Arts and Industries. These are interspersed throughout the volume, and come as a welcome relief to the tedium of a long series of lessons on the metals and their compounds.

Inorganic Chemical Preparations. By Felix Lengfeld, Assistant Professor of Inorganic Chemistry in the University of Chicago. New York: The Macmillan Company. London: Macmillan and Co., Ltd. 1899. Pp. 57.

This manual was written for the classes in Inorganic Preparations at the University of Chicago, and is intended for advanced students who are supposed to be able, at the end of the course, to make any inorganic preparation without assistance from an instructor. The methods given have all been verified, and found to give good results in the hands of a careful student. The student should first read and remember the general directions, and then, with ordinary intelligence, there is no reason why he should not be able to carry out all the methods and processes given; it will be advisable to begin at the beginning, and work steadily through as the preparations are given, not alphabetically or in groups, but in order of increasing difficulty.

At the same time book-work must not be neglected, as practice and theory go hand-in-hand, and mutually help one another. Numerous references to original articles are given throughout, and these should be looked up and studied.

Elementary Practical Chemistry. By Matthew A. Parker, B.Sc., and D. R. Boyd, Ph.D., B.Sc. Glasgow and Edinburgh: William Hodge and Co. 1899. Pp. 82.

The aim of the writers of this book has been to make it essentially a laboratory guide; it was written primarily for the author's own students, and is in most respects very similar to other books for beginners which are so constantly being published. To some extent the plan has been adopted of not fully describing the results to be obtained in a given experiment, but of leaving these to be determined by the student's own observation. We rather doubt the wisdom of this plan; most students require to be told what to expect, otherwise they are liable to be misled, or will not trouble to make sure that their results are what they should be.

A Treatise on Quantitative Electrolytic Chemical Analysis. ("Traité d'Analyse Chimique Quantitative par Electrolyse"). By M. J. Riban. With 96 Illustrations. Paris: Masson et Cie. 1899. Pp. 304.

Every day, quantitative chemical analysis by means of electrolysis acquires more and more importance; by its means delicate problems have been greatly simplified and taken into ordinary use, and, while giving a great increase in rapidity, it has not diminished the necessary accuracy.

The present volume contains the latest progress made in the science, and also gives a *resumé* of the present state of our knowledge of the subject; further, the author not only introduces the reader to electrolytic chemical analysis, but he also gives a practical guide to its daily uses.

The work is divided into four parts. The first part deals with early physical theories, definitions, laws, sources of electricity, measuring apparatus and its use, electrolytic apparatus, &c.; the second part is devoted to the individual estimation of the metals and metalloids by electrolysis; in the third part we find instructions for the separation of the metals one from the other by the same means; while part four is a collection of examples and methods to follow in complex general analyses, more particularly with regard to commercial products and minerals. A number of tables of measures and calculations relating to electrolysis complete the work, which is to be commended for great clearness of expression and style of printing. As is often the case, however, in French books, there is no index.

The Detection of Alkaline Chromates added to Milk as a Preservative.—Alex. Leys.—Fifty c.c. of the milk are evaporated to dryness in a porcelain crucible and calcined at a cherry-red heat; the ash is moistened with concentrated sulphuric acid; if chromates are present the sulphuric acid takes a reddish yellow tint, and a characteristic liberation of reddish fumes of chlorochromic acid is observed.—*Journ. de Pharm.*, x., No. 8.

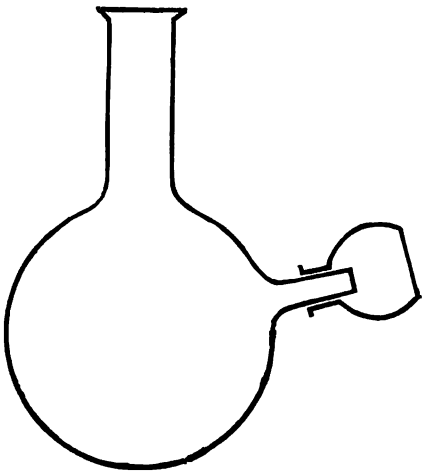
CORRESPONDENCE.

OIL EXTRACTION.

To the Editor of the Chemical News.

Sir,—Having had occasion lately to make a large number of determinations of small quantities of oil, I have been considerably troubled by the difficulty of accurately weighing the comparatively large flasks generally used in extractions; errors, which in milk analysis are of little account, becoming serious when the substance to be weighed only amounts to a few milligrms.

To overcome this difficulty I have devised a special extraction flask, which is clearly shown in the accompanying sketch.



The ordinary flask of about 150 c.c. capacity is provided with a side tube fused on rather above the centre, and on to this side tube is ground a little weighing flask of about 10 c.c. capacity. The side tube projects a little way into the weighing flask, as shown.

The little flask is cleaned, heated in the water-oven, cooled, and weighed. It is then placed in position, and the large flask connected to the extractor as usual. At the close of the extraction the solvent is evaporated off until only enough remains to hold the oil in solution, and the large flask is then disconnected and slowly tilted until the residue runs into the little flask. If the little flask is then warmed, the large flask may be rinsed out by the condensed vapour of the solvent.

When the oil is completely transferred, the little flask is disconnected, heated in the water oven, and weighed.

The flask is of course equally applicable to milk or other extractions where accuracy is desired.—I am, &c.,

Fred. A. Anderson.

73, Queen Victoria Street, E.C.
January 17, 1900.

The Estimation of Persulphates.—G. H. Mondolpho.—This method consists in decomposing the persulphate by means of iodide of potassium, and titrating the iodine set free by means of hyposulphite. Two or 3 grms. of the sample are dissolved in 100 c.c. of water. 10 c.c. of this solution are treated with an excess of iodide of potassium (0·25 to 0·50 grm.), and heated for ten minutes to 60 or 80°. The iodine is then titrated with a decinormal solution of hyposulphite, using starch as an indicator towards the end of the reaction. The proportion of persulphate present is calculated from the equation, $M_2SO_4 + KI = M_2KSO_4 + I$. One c.c. of the hyposulphite solution corresponds to 0·114 grm. of NH_4SO_4, and to 0·0135 grm. of KSO_4.—*Chemiker Zeitung*, xxiii., p. 699.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et de Chimie,
Series 6, Vol. x., No. 4.

New Method for the Formation of Primary Amines.—M. Guerbet.—It has long been known that the nitriles change into amines possessing the same number of atoms of carbon, when treated with nascent hydrogen; the reaction is as follows:—

$$R-CN+4H=R-CH_2-NH_2,$$

R being the monovalent radical. It appeared to the author that the amides might also be able to fix hydrogen, and change into the corresponding amines, losing 1 molecule of water, thus:—

$$R-CO-NH_2+4H=R-CH_2-NH_2+H_2O.$$

This hypothesis has been verified by experiment, and the author has succeeded in partially transforming acetamide into ethylamine, by treating it with nascent hydrogen produced by the action of sodium on boiling amylic alcohol; the reaction being—

$$CH_3-CO-NH_2+4H=CH_3-CH_2-NH_2+H_2O.$$

The method of procedure is as follows:—15 grms. of dry acetamide are dissolved in 600 grms. of amylic alcohol, previously distilled over caustic baryta, to remove all the moisture; 60 grms. of sodium are added, and the whole boiled until all the metal is dissolved. The gas given off, consisting of ammonia, ethylamine, and hydrogen, is passed through hydrochloric acid, which should remain acid till the end; it is then evaporated to dryness, leaving a residue composed of hydrochlorate of ammonium and hydrochlorate of ethylamine, which are easily separated one from the other by absolute alcohol.

On the Purification of Iridium.—E. Leidie.—Already inserted in full.

No. 5.

This number contains no matter of chemical interest.

No. 6.

On Methylnonylketone.—H. Carette.—An acetone obtained by the fractional distillation of essence of rue has been described as methylnonylketone; the same body has also been obtained by the dry distillation of a mixture of cuprate and acetate of lime. With hydroxylamine the author has obtained an oxime which forms the subject of this note; the acetone used was obtained from commercial essence of rue. To obtain it in a state of purity it was combined with bisulphite of ammonia; the crystals thus obtained are insoluble in water, but very soluble in alcohol. Heated with water on the water-bath this bisulphitic compound is decomposed, giving pure methylnonylketone. The boiling-point of this substance at 766 m.m. b.-p. is 230·65°, and at 0·24 m. b.-p. it is 122—123°. The oxime of this acetone was prepared by putting in contact equal molecules of hydrochlorate of hydroxylamine and methylnonylketone,—that is, 21 grms. of hydrochlorate and 51 grms. of acetone; the strictly necessary amount of potash to decompose the hydrochlorate was also added, and a granular mass of butyrous appearance gradually separates. The reaction is greatly accelerated by the addition of enough alcohol to dissolve the acetone. When purified by crystallisation in dilute alcohol, methylnonylketone-oxime appears in the form of elongated prisms, attaining a length of 6 c.m. It is insoluble in water, but soluble in alcohol, ether, benzene, chloroform, and toluene. The crystals are anhydrous and fuse at 46°.

No. 7.

The Bio-chemical Purification of Waters.—M. Tixier.—Among the different substances which have been

proposed for the biological purification of waters, oxidising agents occupy a preponderating place, especially the permanganates. However, the various attempts made in this direction have all been nullified by reason of the impurities introduced by the reagent; for example, the use of permanganate of potash and lime gives rise to the presence of free alkali in the water, and small quantities of free lime (for that is the alkali present in most cases), make the water absolutely unfit for drinking purposes. This difficulty may be completely done away with by using permanganate of alumina and permanganate of baryta in variable proportions. The reactions which take place are as follows:—With waters free from sulphate of lime, for which only a small quantity of baryta would be necessary, we have principally—

$$(Mn_2O_8)_3Al_2 = Al_2O_3 + 3Mn_2O_3 + O_{12}.$$

With waters containing sulphate of lime we get—

$$SO_4Ca + Mn_2O_8Ba = SO_4Ba + Mn_2O_8Ca$$
$$Mn_2O_8Ca + (Mn_2O_8)_3Al_2 = CaOAl_2O_3 + 4Mn_2O_3 + O_{16}.$$

The reactions take place easily in the cold, and the quality of a water can be determined five minutes after the addition of the first drop of the reagent; in some cases the reaction is instantaneous.

No. 8.

Reactions of Argon and Nitrogen on the Mercurial Radicals.—M. Berthelot.—The author's experiments were carried out under the influence of the electric discharge. Dimethyl-mercury, $(CH_3)_2Hg$, in the presence of argon decomposes, forming hydrogen, formene (or ethane, C_2H_6), and mercury. Analysis shows that no trace of argon was absorbed; on the contrary, the dimethyl-mercury absorbs nitrogen; 0·2576 grm. of dimethyl-mercury was operated on in a small bulb, the C_2H_6Hg gave a gas, $H_2+\frac{1}{2}CH_4$, with the absorption of $0·22N_2$; the composition of the condensed matter was therefore $1\frac{3}{4}C+H_3+0·44N$, that is $C_2H_{3·4}N^{0·40}$. Diphenyl-mercury does not easily lend itself to experiment on account of its solid state and the absence of any appreciable vapour tension at the ordinary temperature. However, after being submitted to the action of the electric discharge for twenty-three hours, the author found that 5·1 hundredths of the volume of argon was absorbed. The experiment was repeated on the gaseous residue with a fresh quantity of diphenyl-mercury, and after eighteen hours a further 3·8 hundredths were found to be absorbed.

Comparative Examination of some Rapid Methods for the Purification of Waters.—F. Malmejac.—This paper deals rather with mechanical than chemical action in the purification of water. The author has experimented on four processes, two using alum (Babés and Wernei), and two using perchloride of iron (Almen d'Upsal and Manget). These processes are simple and rapid; the experiments show that the perchloride of iron processes give the best results, as they diminish the proportion of organic matter, either in an acid or alkaline medium, by 60 per cent, while the alum processes only reduce it by 25 and 33 per cent respectively. The two latter, however, have a stronger action—nearly three times as powerful—in destroying microbes.

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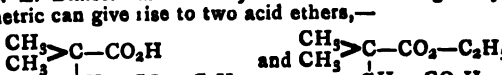
Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxi., No. 15.

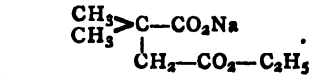
Decomposition of Carbonic Oxide in the presence of Metallic Oxides.—O. Boudouard.—Already inserted.

Decomposition of Carbonic Acid in the presence of Carbon.—O. Boudouard.—Already inserted.

The Synthesis of $\beta\beta$-Dimethyl-levulic Acid.—E. E. Blaise.—$\alpha\alpha$-Dimethylsuccinic acid being dissymmetric can give rise to two acid ethers,—

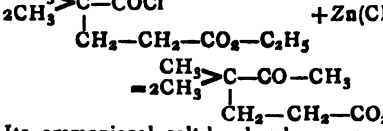


To obtain $\beta\beta$-dimethyl-levulic acid, which is the same body as is formed by the oxidation of campholene, we must start with the first of these two acids. The author finds that the anhydride corresponding to $\alpha\alpha$-dimethylsuccinic acid, treated with ethylate of sodium, gives an ether salt,—



He points out that while in the case of succinic anhydride a notable quantity of disodic succinate is precipitated, in that of its dimethyl derivative only traces of the neutral salt are formed. 30 grms. of the preceding acid ether and 26 grms. of trichloride of phosphorus are placed in a flask fitted with a vertical condenser, and heated gently on the water-bath at 60—70° for 45 minutes. On cooling, the chloride ether of phosphorus anhydride is separated by decantation. Dimethyl-levulic acid, $CH_3—CO—C(CH_3)_2—CH_2—CO_2H$, is obtained by the action of zinc methyl on the preceding chloride ether. The product of the reaction is decomposed by water, and gives a benzenic solution from which the carbide is separated by distillation. The residue is fractionated *in vacuo*; the portion passing between 105—110° at 20 m.m. is rectified, and then boils at 106—107°. This body is dimethyl-levulic ether; it is saponified with potash, acidulated with hydrochloric acid, and evaporated to dryness; the residue exhausted with ether yields to this solvent dimethyl-levulic acid, which may be purified by distillation *in vacuo*.

The Synthesis of $\gamma\gamma$-Dimethylhexanonoïc Acid, $CH_3—CO—C(CH_3)_2—CH_2—CH_2—CO_2H$.—E. E. Blaise,—This acid has been obtained by the action of zinc-methyl on the chloride ether of $\alpha\alpha$- dimethylglutaric acid,



Its ammoniacal salt has has been prepared by passing a current of ammonia gas through an ethereal solution of the acid. It separates in the form of a crystalline precipitate, which when dry melts at 115—118°.

The Preparation of the Phenolic Chloro-carbonates.—E. Barral and A. Morel.—The authors have studied the action of $COCl_2$ on the phenols with two different objects: (1) to obtain neutral carbonates of chlorophenols, and to identify them with the products of direct chlorinisation of the carbonate of benzenol; and (2) to study the action of alcohols on the phenolic chloro-carbonates after having effected the same on the neutral phenolic carbonates and phosphates. They found that the more concentrated the alkaline phenate is, the more neutral carbonate is there formed; at too low a temperature the action does not take place; at too high a temperature (above 50°) the oxychloride decomposes, and with the most favourable conditions of concentration and temperature it is impossible to prevent the formation of the alkaline carbonate when using certain phenates, such as those of the naphthols and diphenols. In such cases the phenol is set at liberty, and very little symmetric carcarbonate and chloro-carbonate is formed.

On some Phenolic Chloro-carbonates.—E. Barral and A. Morel.—The authors have prepared the chloro-carbonates or chloroformates of benzenol, para-cresol, thymol, and gaïacol, and they here describe their properties.

Action of Formic Aldehyd on Albumenoid Matters.—C. Lepierre.—The author finds that the *proto-albumoses* are rendered insoluble by warm CH_2O; that the *dextro-albumoses* are not homogeneous bodies, but a collection of bodies formed of homologous units, the first of which, of a high molecular weight, are nearest to the proto-albumoses, and are rendered insoluble by CH_2O;

the others, nearer the true peptones, are simply transformed into proto-albumoses, and are changed into insoluble derivatives by prolonged action of the reagent; and (3) the true peptones are first transformed into bodies of the dextro-class, and these again into proto-albumoses.

Cryoscopy of Butters and Margarines.—M. Pouret.—This method of examining butters for adulteration, though giving certain indications, is not sufficiently exact for practical purposes, the composition of different butters being so variable; in some cases as much as 15 per cent of margarine could be added without detection.

MISCELLANEOUS

A New Method for the Determination of Nitric Acid.—J. F. Pool.—The following method, according to the author, gives exact results for the estimation of nitric acid in potable waters, manures, &c. The sample is evaporated to dryness in an Erlenmeyer flask with an excess of chloride of sodium. The dry mass is decomposed by sulphuric acid in an atmosphere of carbonic acid, the mixture is diluted with water, and the chlorine driven off by boiling. The fumes given off are collected in a solution of iodide of potassium, and the iodine set at liberty is determined by titration. By operating, as has been said, in an atmosphere of carbonic acid, the binoxide of nitrogen set free according to the equation, $6HCl+2HNO_3=2NO+4H_2O+3Cl_2$, is not transformed by oxidation, into products also capable of liberating iodine and passing into solution in the iodide of potassium.—*Nederl. Tijdsch. Pharm.*, xi., 171.

The use of Alkaline Solutions of Formic Aldehyde in Quantitative Analysis.—L. Vanino.—Gold can be estimated quantitatively by adding ordinary commercial formalin to a solution of the chloride, a few drops of caustic soda are then added and the whole heated for a few moments on the water-bath. The metallic gold is separated by filtration, and the filtered liquid is tested again by formalin to make sure that the precipitation is complete. The precipitated gold is washed with water, and then alcohol, and dried at 180°, or even heated to redness in a crucible. Silver can also be precipitated from its solution of nitrate by formalin, either in the cold or on a water-bath. In the latter case it is essential to wash the precipitate with absolute alcohol to remove the adhering water. Chloride of silver is reduced by formalin. This reaction can be applied, not only to quantitative analysis, but also to the preparation of pure silver from silver residues.—*Berichte*, xxxi. (II.), p. 1763.

Institute of Chemistry Examinations.—Pass Lists (January, 1900).—*Intermediate Examination*: F. W. Aston, Mason Univ. Coll., Birmingham; J. H. Austin, Mason Univ. Coll., Birmingham; H. J. Bailey, Univ. Coll., Sheffield; H. G. Bayly, King's Coll., London; G. Clarke, jun., Univ. Coll., Nottingham; M. W. Danks, Mason Univ. Coll., Birmingham: R. E. Blake Smith, B.Sc. (Lond.), Univ. Coll., London; Thomas Taylor, Glasgow and W. of Scotland Tech. Coll.; John Webster, Mason Univ. Coll., Birmingham; and W. E. Woodman, King's Coll., London. *A.I.C. Examination* (Old Regulations): J. R. Brooke, Pharmaceutical Society's Laboratories and King's Coll., London; M. Priest, Finsbury Tech. Coll.; and T. Tickle, Pharmaceutical Society's Laboratories, King's Coll. and Univ. Coll. London. *Final A.I.C. Examination* (*Branch* "A," *Mineral Chemistry*): W. A. Fyffe, Univ. Coll., Dundee; and L. V. Wright, B.A., Sidney Coll., Cambridge. *Branch* "B" (*Metallurgical Chemistry*): H. J. Winch, A.C.G.I., City and Guilds of London Central Institution and Finsbury Tech. Coll. *Branch* "C" (*Physical Chemistry*): T. S. Price, B.Sc. (Lond.), Ph.D. (Leipzig), Mason Univ. Coll., Birmingham, and the Universities of Leipzig and Stockholm. *Branch* "D" (*Organic Chemistry*): F. G. Edmed, Assoc.R.C.Sc. (Lond.), B.Sc., (Lond.), Royal College of Science, London; L. Eynon, Finsbury Tech. Coll.; H. Hall, Univ. Coll., Nottingham, and F. Shedden, B.Sc. (Lond.), Mason Univ. Coll., Birmingham. *Branch* "E" (*the Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology, and Microscopy*): L. W. Stansell, Maidstone, and W. Thorp, B.Sc. (Lond.), Limerick, both for the Fellowship. The Examiners in Chemistry were Dr. Bernard Dyer, D.Sc. (Lond.), F.I.C., and Professor Percy F. Frankland, F.R.S., F.I.C.., and the Examiner in Therapeutics, Pharmacology, and Microscopy was Dr. Thomas Stevenson, M.D., F.R.C.P.

NOTES AND QUERIES.

Water Analyses.—I have had a sample of water submitted to me for analysis, and have obtained the following results, in parts per 100,000:—

Total solids..	62·0
Sulphate of lime	40·2
Chlorine	2·4
Free ammonia	0·001
Albumenoid ammonia	0·0034
Poisonous metals	nil

Can any reader inform me if this water may be pronounced fit for drinking purposes, or is the sulphate of lime excessive? The books and journals which I have access to do not help me to decide the matter. Information on the subject will be gratefully received.—N. H.

MEETINGS FOR THE WEEK.

Monday, 29th.—Society of Arts, 8. (Cantor Lectures). "The Nature and Yield of Metalliferous Deposits," by Bennett H. Brough.

Tuesday, 30th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester M.A., LL.D., F.R.S.

—— Society of Arts, 8. "Niello Work," by Cyril Davenport.

Wednesday, 31st.—Society of Arts, 8. "The Undeveloped Resources of the Bolivian Andes," by Sir Martin Conway, M.A.

Thursday, Feb 1st.—Royal Institution, 3. "The Senses of Primitive Man," by W. H. R. Rivers, M.A., M.D.

—— Chemical, 8. "The Chlorine Derivatives of Pyridine—Part V. Synthesis of $\alpha\alpha'$-Dichloropyridine and Constitution of Citrazinic Acid," by W. J. Sell, M.A., and F. W. Dootson, M.A. "The Formation of Heterocyclic Compounds," by S. Ruhemann and H. E. Stapleton. "The Space Configuration of Quadrivalent Sulphur Derivatives—Methylethylthetin-dextrocamphorsulphonate and Dextrobromocamphorsulphonate," by W. J. Pope and S. J. Peachey. "Nitrocamphane," by M. O. Forster.

Friday, 2nd.—Royal Institution, 9. "Wireless Telegraphy," by G. Marconi, M.Inst.E.E.

Saturday, 3rd.—Royal Institution, 3. "Neglected Byways in Music" (with Musical Illustrations), by Sir Hubert H. Parry, Mus. Doc., M.A., D.C.L.

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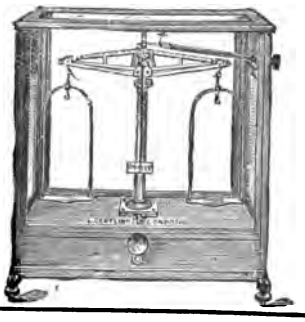

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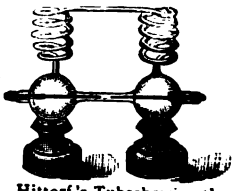

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THE CHEMICAL NEWS.

VOL. LXXXI., No. 2097.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART II.

By HARRY BREARLEY.

(Continued from vol. lxxx., p. 273).

TUNGSTEN AND MOLYBDENUM.

THIS section is compiled from the following journals:—

CHEMICAL NEWS, 1860 to 1899, vols. i. to lxxx.
Journal of the Iron and Steel Institute, 1880 to 1899.
Journal of the Chemical Society, 1885 to 1898, vols. xlvii. to lxxiv.
Journal of the Society of Chemical Industry, 1882 to 1898, vols. i. to xvii.

The general arrangement for both elements alike is—

I. SEPARATION FROM OTHER ELEMENTS.
II. GRAVIMETRIC ESTIMATIONS.
III. VOLUMETRIC ESTIMATIONS.
IV. DETECTION AND MISCELLANEOUS NOTES.

The analytical characters of the two metals are so similar that the means of separating or estimating the one frequently apply to the other also. Attention is drawn to such facts when they occur, but they are not separately mentioned under each element.

Tungsten having come into general use within the last dozen years or so, and molybdenum being hardly in general use even now, the analysis of steel works compounds of these metals has no very extensive literature. On this account, references to the analysis of compounds foreign to steel works practice are included in order that the processes for the one may, perhaps, be adapted to the other material.

Special attention is drawn to the fact that SiO_2 and WO_3 cannot be separated by ammonia. Dufty, in a paper read before the Sheffield Metallurgical Society, seems first to have drawn attention to this fact, but Skey and Pibram had previously (219) observed the solubility of even ignited silica in ammonia.

Some references, chiefly to volumetric processes of estimating molybdenum, are properly associated with the molybdate process of estimating phosphorus.

TUNGSTEN.

I. SEPARATION FROM OTHER ELEMENTS.

218. *Iron.*—BENNEVILLE (*J. I. S. I.*, 1895, i., 205).—Fuse with Na_2CO_3, KNO_3, and NaHO; extract with water, and evaporate with HCl. Fe and Mo are also separated by fusion. (See 258 and 259).

219. *Silicon.*—SKEY (*C. N.*, xvii., 165).—Hydrous SiO_2 is sensibly dissolved in ammonia, and is partly soluble even after strong ignition. PIBRAM (*C. N.*, xvii., 227) determines the solubility of variously treated silica in ammonia.

o VIGOUROUX (*C. N.*, lxxviii., 291).—Silicide of tungsten is analysed by heating with chlorine. The silicon chloride is volatilised and finally weighed as SiO_2.

221. WADDELL (*C. N.*, l., 113).—By fusion with $KHSO_4$, &c.; also with ammonia.

222. BENNEVILLE (*J. S. C. I.*, 1897, 636).—Several evaporations with HF are sometimes necessary to remove all the SiO_2 from WO_3. Accurate separation with ammonia not possible.

223. *Tin.*—RAMMELSBERG (*C. N.*, ix., 25).—Rose's process consists in reducing the metals in hydrogen, and dissolving out the tin with HCl (see 274). It is difficult to reduce the WO_3, and the tin may volatilise. By igniting with $(NH_4)Cl$ stannic acid is vapourised; WO_3 remains. Repeat process to constant weight (see 279).

224. TALBOTT (*C. N.*, xxii., 190).—Fuse oxides with KCN. The tin is reduced to metal and filtered from alkaline tungstate dissolved in water. Mo is not similarly separated.

225. DONATH and MÜLLNER (*J. C. S.*, liv., 531).—Oxides mixed with zinc dust, ignited, boiled with HCl, oxidised with $KClO_3$, and WO_3 filtered off.

226. *Phosphorus.*—KEHRMANN (*J. I. S. I.*, 1887, ii., 371).—Boil acids with NaHO; $(NH_4)Cl$ and $(NH_4)HO$ added, and the P precipitated with magnesia mixture. Filtrate must be evaporated four times to recover the WO_3.

227. *Vanadium.*—FRIEDHEIM (*C. N.*, lxi., 220).—The acids are precipitated together with $HgNO_3$ and HgO, and moist precipitate dissolved in HCl; on largely diluting, the WO_3 is re-precipitated and separated by filtration.

228. GIBBS (*J. C. S.*, xlvi., 713).—Slightly alkaline solution boiled with large quantity of $(NH_4)NO_3$; metavanadate precipitated on standing. ROSENHEIM (*J. C. S.*, lvi., 762) says Gibbs's process is erroneous.

229. ROTHENBACH (*J. C. S.*, lx., 18).—The acids precipitated together with $HgNO_3$; the vanadic acid determined separately by reduction and titration with $KMnO_4$, or by boiling with H_3PO_4, KBr, and HCl, absorbing liberated Br in KI, and titrating the free iodine.

230. BROWNING and GOODMAN (*J. C. S.*, lxxii., 234).—Va can be estimated in the presence of both W and Mo by reducing with some organic acid, and titrating with iodine (see also 281).

231. *Titanium.*—DEFACQZ (*C. N.*, lxxiv., 293).—Heated with KNO_3 and K_2CO_3; W in water extract; precipitate as mercurous tungstate.

232. *Gold.*—SMITH and WALLACE (*J. C. S.*, lxii., 920).—Both W and Mo are separated electrolytically.

233. *Molybdenum.*—FRIEDHEIM and MEYER (*J. C. S.*, lxiv., 125).—Tungsten compounds always contain molybdenum; it is separated as sulphide from a solution of the metatungstate.

234. BENNEVILLE (*J. I. S. I.*, 1895, i., 209).—Precipitated together as lead salts, heated in stream of HCl, residual tungsten determined, and Mo calculated by difference.

235. SMITH and OBERHOLTZER (*C. N.*, lxviii., 6).—By volatilising Mo in current of HCl. SMITH and MAAS (*C. N.*, lxviii., 207) make an atomic weight determination in this way.

236. WADDELL (*C. N.*, lv., 101).—Add tartaric acid to prevent precipitation of WO_3 on acidification; the Mo is then precipitated with H_2S.

237. DESI (*J. C. S.*, lxxiv., ii., 231).—WO_3 insoluble in H_2SO_4; MoO_3 easily dissolved; hence a means of separating them (see also 287).

238. *Antimony.*—HALLOPEAU (*J. C. S.*, lxxiv., ii., 540).—The metals are precipitated as mercurous antimonio-tungstate, ignited to oxides, and the antimony reduced by fusion with KCN.

239. *Antimony, Arsenic, and Iron.*—COBENZL (*C. N.*, xlvi., 13).—Digest with aqua regia, evaporate, take up with HNO_3 and tartaric acid, and filter off WO_3 (see 280).

240. *Silver, Mercury, and Cadmium.*—SMITH and FRANKEL (*J. S. C. I.*, 1890, 1067).—Separated electrolytically from both W and Mo.

240*a*. *Chromium.*—SMITH and DIECK (*C. N.*, lxviii., 68).—Evaporate with HCl. Pure WO_3 remains insoluble on again digesting with HCl. (See 263).

240*b*. *Various.*—Ibbotson and Brearley (*C. N.*, lxxx., 293).—The alkalis, alkaline earths, Zn, Cd, Mn, Ni, Co, Hg, As, Ur, and Fe do not interfere with the precipitation of W as $PbWO_4$ followed by solution in HCl, and precipitation of WO_3 by diluting.

(To be continued).

A CONTRIBUTION TO THE STUDY OF LIQUID MIXTURES OF CONSTANT BOILING-POINT.*

By GARNETT RYLAND.

(Concluded from p. 43).

General Investigation of the Boiling-points of Binary Mixtures of Liquids Mutually Soluble in all Proportions.

All liquids boiling below 170° that were at hand were purified; and each was mixed with every other whose boiling-point was within 30—40° of its own, and the mixture distilled. When a constant boiling mixture was found, the ratio of its constituents was determined as nearly as possible by repeated mixing in various proportions, and careful distillation until that ratio was discovered for which the whole mixture distilled most nearly within 1°. A volume of 10—15 c.c. was found adequate for each trial mixture distilled.

The ratio of the products of the respective vapour-densities and vapour-tensions at a temperature near that of constant boiling was calculated whenever the necessary data were known, and is given below as the "theoretical ratio."

1. Methyl alcohol (64·5—65°) and ethyl bromide (37·5—38·5°) are distilled at 35—36° (765 m.m.) in the approximate ratio of 5 : 95 parts by weight. Theoretical ratio at 35°, 9 : 91 parts by weight.

2. Methyl alcohol and chloroform (60—61°) at 53·5—54·5° (765 m.m.); ratio, 12 : 88. Theoretical ratio at 55°, 17 : 83.

3. Methyl alcohol and methyl acetate (55·5—56·5°) at 53·5—54·5° (760 m.m.); ratio, 18 : 82.

4. Methyl alcohol and ethyl iodide (see Wanklyn p. 16, *ante*) (72·3—72·5°) at 54·5—55·5° (770 m.m.); ratio, 17 : 83. Theoretical ratio at 55°, 19 : 81.

5. Methyl alcohol and ethyl acetate (75·5—76·5°) at 61·7—62·5° (757 m.m.); ratio, 47 : 53.

6. Methyl alcohol and benzene. (See p. 42, *ante*).

7. Methyl alcohol and isobutyl iodide (118—119°) at 63·5—64·5° (760 m.m.). The ratio of 70 : 30 parts by weight gave an excess of alcohol. In this and all succeeding distillations of isobutyl iodide mixtures, the formation of tar made a close determination of the proportions impossible.

Methyl alcohol is separated by fractional distillation from ether (34·5—35·5°), acetone (56—57°), and toluene (108·5—109·3°).

8. Ethyl alcohol (77·5—78°) and ethyl bromide 37·5—38·5°) at 36·5—37·5° (762 m.m.). The theoretical ratio at 37·5° is 6·6 : 93·4. The actual per cent of alcohol seemed to be about half of the theoretical, but could not be more closely determined.

9. Ethyl alcohol and carbon disulphide (see Berthelot, p. 16, *ante*) (45·5—46°) at 41·5—42·5° (755 m.m.); ratio, 9 : 91, verified. Theoretical ratio at 43—44°, 11·4 : 88·6.

10. Ethyl alcohol and chloroform (60—61°) at 58·5—59·5° (759 m.m.); ratio, 6 : 94. Theoretical ratio at 60°, 15·6 : 84·4.

11. Ethyl alcohol and ethyl iodide (71·5—72·5°) at 62·5—63·5° (761 m.m.); ratio, 14 : 86. Theoretical ratio at 60°, 17·4 : 82·6.

12. Ethyl alcohol and ethyl acetate (75·5—76·5°) at 71—72° (765 m.m.); ratio, 31 : 69.

13. Ethyl alcohol and benzene. (See p. 42, *ante*).

14. Ethyl alcohol and isobutyl iodide (118—119°) at 76·5—77·5° (760 m.m.); approximate ratio, 70 : 30.

Ethyl alcohol is separated from propyl alcohol (97·5°) and toluene (108·7—109·3°).

15. Isopropyl alcohol (81—82°) and carbon disulphide 45·5—46°) at 43·5—44·5° (761 m.m.); ratio, 9 : 91.

16. Isopropyl alcohol and ethyl iodide (71·5—72·5°) at 65·5—66·5° (764 m.m.); ratio, 13 : 87.

17. Isopropyl alcohol and ethyl acetate (75·5—76·5°) at 74—75° (770 m.m.); approximate ratio, 26 : 74.

18. Isopropyl alcohol and benzene (79—79·5°) at 71—72° (758 m.m.); ratio, 30 : 70.

19. Isopropyl alcohol and water at 78·5—79·5° (768 m.m.); ratio, 89 : 11.

(See Linnemann, p. 16, *ante*. The per cent of water in the compounds $3C_3H_8O.2H_2O$, $2C_3H_8O.H_2O$, and $3C_3H_8O.H_2O$ is 16·7, 13, and 9 per cent respectively. The alcohol used by me was distilled from lime at 81—82°, This case should be investigated further).

20. Isopropyl alcohol and isobutyl iodide (118—119°) at 81—82° (761 m.m.); approximate ratio, 70 : 30.

Isopropyl alcohol is separated from chloroform (60—61°) and toluene (108·7—109 3°).

21. Normal propyl alcohol (95·7°) and ethyl iodide (72·3—72·5°) at 69·5—70·5° (768 m.m.); approximate ratio, 7 : 93.

22. Propyl alcohol and benzene (79—79 5°) at 76—77° (762 m.m.); ratio, 16·5 : 83·5. Theoretical ratio at 80°, 28·5 : 71·5.

23. Propyl alcohol and water at 87—87·5° (770 m.m.); ratio, 72 : 28.* Theoretical ratio at 87·5°, 79 : 21.

24. Propyl alcohol and toluene (108·7—109·3°) at 91—92° (765 m.m.); ratio, 53 : 47.

25. Propyl alcohol and isobutyl iodide (118—119°) at 92·5—93·5° (753 m.m.); approximate ratio, 45 : 55.

Normal propyl alcohol is separated from ethyl acetate (75·5—76·5°) and isobutyl alcohol (105·3—106·3°).

26. Isobutyl alcohol (105·3—106·3°) and toluene 108·8—109·3°) at 100° (764 m.m.); ratio, 43 : 57.

27. Isobutyl alcohol and isobutyl iodide (118—119°) at 101—102° (765 m.m.). The ratio of 33 : 67 gave an excess of alcohol.

28. Isobutyl alcohol and ethylene bromide (129—130° at 104·5—105·5° (763 m.m.); approximate ratio, 62 : 38.

Isobutyl alcohol is separated from ethyl iodide (72·3—72·5°), benzene (78·5—79°), and metaxylene (136—137°).

29. Amyl alcohol (128—129°)† and isobutyl iodide (118—119°) at 115—116° (765 m.m.). The ratio of 20 : 80 gave a slight excess of iodide.

30. Amyl alcohol and ethylene bromide (129—130°) at 121—122° (760 m.m.); ratio, 30 : 70. Ratio of vapour-densities, 32 : 68.

31. Amyl alcohol and metaxylene (136—137°) at 125—126° (760 m.m.); ratio, 52 : 48. Amyl alcohol and paraxylene (137—137·5°) at 125—126°, in the same ratio; and amyl alcohol and orthoxylene (140—141°) at 127—128°, with a slightly larger per cent of alcohol.

Amyl alcohol is separated from toluene (108·8—109·3°).

32. Allyl alcohol (95—96°) and benzene (79—79·5°) at 76—77° (760 m.m.). The ratio of 20 : 80 gave a slight excess of alcohol.

33. Allyl alcohol and toluene (108·8—109·3°) at 91—92° (756 m.m.); approximate ratio 50 : 50.

34. Acetic acid (117—118°) and toluene (108·8—109·3°) at 103·5—104·5° (761 m.m.); ratio, 30 : 70.

35. Acetic acid and metaxylene (136—137°) at 113·5—114·5° (761 m.m.); ratio, 27 : 73.

Acetic acid is separated from carbon disulphide (45·5—45·7°) and benzene (79—79·5°).

36. Butyric acid (159—160°) and water at 99—99·5°

* From the *American Chemical Journal*, vol. xxii., No. 5, Nov., 1899.

* See Konowalow, and Ramsay and Young, p. 16 (*ante*). My determination of the ratio was made before I knew of that found by Ramsay and Young.

† This was obtained by repeated fractionation from the commercial mixture of amyl alcohols.

(763 m.m.); ratio, 20 : 80 (see Konowalow, p. 16, *ante*). Theoretical ratio at 100°, 32 : 68.

37. Butyric acid and brombenzene (152—153°) at 147—148° (748 m.m.); ratio, 19 : 81.

38. Ether (34·5—35·5°) and carbon disulphide (45·5—46°) at 34·5—35·5° (768 m.m.); approximate ratio, 65 : 35. Theoretical ratio at 35°, 59 : 41.

Ether is separated from acetone (56—57°) and chloroform (60—61°). A mixture of ether and ethyl bromide (37·5—38·5°) is distilled at the same temperature (35—38°) as its constituents.

39. Carbon disulphide (45·5—45·7°) and ethyl bromide (37·5—38·5°) at 37—38° (770 m.m.); ratio, 32 : 68. Theoretical ratio at 37·5°, 35 : 65.

40. Carbon disulphide and acetone (55·5—56·5°) at 38·5—39·5° (766 m.m.); ratio, 74 : 26. Theoretical ratio at 40°, 66 : 34

41. Carbon disulphide and methyl acetate (55·5—56·5°) at 39—40° (756 m.m.); ratio, 71 : 29.

42. Carbon disulphide and ethyl acetate (75·5—76·5) at 45·5—46 5° (768 m.m.); ratio, 92 : 8.

Carbon disulphide is separated from chloroform (60—61°), ethyl iodide (71·5—72·5°), and benzene (79—79·5°).

43. Acetone (55·5—56·5°) and methyl acetate (55·5—56·5°) in about equal parts are distilled 0·5° below the boiling-point of either.

44. Acetone and ethyl iodide (71°5—72·5°) at 55—56° (770 m.m.); approximate ratio, 60 : 40.

45. Acetone and chloroform (60—61°) at 64—65° (760 m.m.); approximate ratio, 20 : 80.

Acetone is separated from ethyl bromide (37·5—38·5°) and ethyl acetate (75·5—76·5°).

46. Chloroform (60—61°), and methyl acetate (55·5—56·5°) at 64—65° (760 m.m.); approximate ratio, 78 : 22.

Chloroform is separated from ethyl bromide (37·5—38·5°), ethyl iodide (71·5—72·5°), ethyl acetate (76—77°), and benzene (79—79·5°).

47. Ethyl iodide (71·5—72·5°), and ethyl acetate (75 5—76·5°), at 69·5—70·5° (762 m.m.); approximate ratio, 78 : 22.

48. Ethyl iodide and benzene (79—79·5°) at 74—75° (758 m.m.); approximate ratio, 80 : 20.

Ethyl acetate (75·5—76·5°) is separated from benzene (79—79·5°).

Isobutyl iodide (118—119°) is separated from toluene (108·7—109·3°).

Acetone (55·5—56·5°), and benzene (79—79·5°) in the ratio of 5 : 1 approximately, showed a tendency to gather at 57—58°, this fraction constantly increasing throughout the course of repeated distillations. But the proportions could not be found for which all of the mixture would boil at this point, as in every case there were small fractions from 57° to 79°.

Ethylene bromide (129—130°) and metaxylene (136—137°), in the ratio of 5 : 2 approximately, showed a similar tendency to gather at 130—131°.

Amyl alcohol (128—129°), and brombenzene (152—153°), in the ratio of 5 : 2 approximately, seemed to be distilled at 128—129°, but the small per cent of the latter and the formation of tar prevented a definite determination.

Summary.—Of the 80 mixtures examined, 45 were distilled at a constant temperature at or below the boiling-point of the more volatile constituent; 2 (Nos. 45 and 46), at a constant temperature above the boiling-po nt of the less volatile constituent; 1 (Nos. 48), at a constant temperature between the boiling points of the constituents; 1 (ether-ethyl bromide), in which the boiling-points of the constituents are too close for fractionation, presents no relative depression or elevation of the boiling-point of the mixture; and 3 are of an uncertain character.

Boiling-points of Mixtures of Liquids whose Mutual Solubilities are Limited.

Two cases of this kind were examined.

1. Isobutyl alcohol (105·3—106·3°), saturated with water at the temperature of the room (about 5 parts of alcohol to one part of water) boils for a time at the constant temperature of 89—90°, the distillate separating in the receiver into two layers. The excess of alcohol above the amount necessary for the proportions of distillation at constant temperature causes the temperature to rise gradually to 106°. If the alcohol is saturated at higher temperatures, the portion distilling at 89—90° is correspondingly larger.

2. Methyl alcohol (64·5—65°), saturated with carbon disulphide (45·5—46°) (about equal parts by weight at the temperature of the room), boils for a time at 37—38°. The excess of alcohol causes the temperature to rise ultimately to 65°. This mixture shows phenomena similar to those noted in case 1.

Conclusions.

1. The large proportion of constant boiling mixtures found in the cases examined makes it evident that this phenomenon is by no means so exceptional as has hitherto been supposed. In fractional distillation one should always be on his guard against it.

2. The changes in the composition of the distillates, corresponding to the changes in pressure and temperature of the distillation, indicate that the two mixtures investigated (see p. 42, *ante*), are not true chemical compounds, and that any approximation to molecular proportions must be regarded as accidental.

3. It is well known that two liquids mutually insoluble are distilled at a constant temperature in the ratio of the products of their respective vapour-densities and vapour-tensions at the temperature of distillation.

If the liquids are partially soluble in each other, they are also distilled at a constant temperature, but with some deviation from this ratio, so long as there are two layers of liquids in the distilling flask.

The results of this investigation are generally in accord with the theory that when two liquids, mutually soluble in all proportions, are distilled at a constant temperature, their ratio in the distillate is that of the products of the vapour-densities and vapour-tensions of the pure liquids at that temperature, modified more or less by their influence upon each other.*

ON GADOLINIUM.†

By C. BENEDICKS.

Introduction.

THE study of the so-called rare-earths was pursued in a new direction in the year 1873, on the appearance of Cleve and Höglund's work on the salts of yttrium and erbium. Hitherto the experimenters whose names are connected with the history of the rare earths had specially devoted their attention to the pure production of a certain oxide, in order to determine its atomic weight, the phenomena exhibited by its spectrum, and the common properties of the salts. The first result of such experiments was the establishment of the tri-atomicity of these elements. It is clear that in this direction we may expect other important results for this interesting, but obscure, field of chemical research, at least so soon as similar material is acquired from a larger number of more closely examined earths. With reference to this, Prof. Dr. P. T. Cleve has requested me to work upon the salts of gadolinium. In the following I shall report upon this work, and shall also mention what is so far known concerning this element.

I take this opportunity of acknowledging my gratitude to Prof. Dr. P. T. Cleve for the interest and advice he has accorded me during the work.

* The greatest deviation from the calculated ratio was found in the mixture of propyl alcohol and benzene (No. 22).

† *Zeitschrift für Anorganische Chemie*, xxii., 5, p. 393.

Historical.

In the year 1880, Marignac discovered a new earth in samarskite—a mineral which, two years before, had been found in great quantity in North Carolina. He succeeded in isolating two different oxides which were formerly called by him Yα and Yβ. Yβ, which was identified by the yellow colour and the absorption spectrum of its salts, was later found to be identical with the oxide of samarium, discovered by Lecoq de Boisbaudran in 1879.

Marignac, with his well-known accuracy, ascribed to the earth Yα, which had no absorption spectrum, and whose salt solutions were colourless, properties which at once characterise it as the oxide of a new element. I quote the following extract from his work:—"The existence of this base as a separate earth is made evident by the fact that its equivalent is a maximum between those which come next to it by their solubility in potassium sulphate." . . . "It should not be confounded with any one of those so far identified."

The solubility in saturated potassium sulphate solution, which divides Yα, on the one hand, from easily soluble ytterbium and terbium oxides, and, on the other hand, from difficultly soluble samarium oxide (Yβ), was stated by Marignac to be such that 1 grm. oxide was held in solution as double sulphate of potassium by 100—150 c.c. potassium sulphate solution; and this statement was later fully corroborated (by Bettendorff and me). The atomic weight was found by Marignac to be 156·75 (equivalent weight $\frac{II}{2}O = 120·5$), which agrees pretty closely with the later found values.

But, according to Marignac, it was possibly that his earth Yα was identical with philippium, found shortly before by Delafontaine. Added to this, mosandrium, identified by Law. Smith, was still doubtful, though looked upon by Marignac and Delafontaine as terbium, and he hesitated for the present to give his earth a name. He writes:—"I do not consider it yet necessary to give a name to this new earth. It will be time enough when one is certain of having obtained it in a state of purity, and succeeded in preparing its salts in sufficient quantity for examination."

Afterwards, Marignac's work was continued by Lecoq de Boisbaudran, by successive fractionation with dilute ammonia. The atomic weight determinations carried out by him, and which agreed well with those of Marignac, have placed the individuality of Yα beyond doubt. After he had proved mosandrium to be a mixture of Yα and terbium, he, with the concurrence of Marignac, fixed for Yα in 1886 the name gadolinium, with the symbol Gd.

It is certainly very questionable if this name is appropriate for a fundamental substance, in conformity with the usual practice in chemistry not to derive names of so much importance as those of the fundamental substances from persons, however eminent. The name is derived from the Finnish chemist Gadolin, who, in 1794, discovered the mineral gadolinite, afterwards named after him.

However, the same remark might be made with reference to the name samarium (the mineral samarskite is called after the Russian v. Samarski), and as the name gadolinium is already enrolled in literature, it might not be advisable to replace it by another.

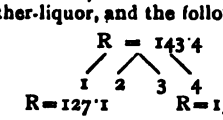
In the year 1892 Bettendorff produced a pure specimen of gadolinium earth. He carried out atomic weight determinations in various potassium sulphate fractions, from which it appeared that in these there was no variation of the atomic weight. Also, by partial precipitation with ammonia, an earth with a quite unaltered atomic weight was obtained, and by this Bettendorff established the identity of gadolinium.

A new method of fractionating was proposed in 1896, when Demarçay crystallised a mixture of gadolinium and samarium nitrates in concentrated nitric acid, and expected to find in the fraction between gadolinium (which crystallises first) and samarium (which remains more in the mother-liquor) a new element, distinguishable by its spark spectrum from the above-mentioned and remaining earth elements. In the following I will give a fuller description of this.

Methods of Separation and Material.

For the isolation of gadolinium from accompanying earths, Marignac made use of the difference in solubility in potassium sulphate solution, by which gadolinium is distinguished from the more soluble yttrium and terbium, and the less soluble samarium (and didymium); this method is tedious from much repetition. It must be combined with partial precipitation by ammonia, in order to obtain a pure product, especially in the case of separating terbium. By this means, terbium and samarium are precipitated before gadolinium, and didymium after gadolinium. The material kindly placed at my disposal by Professor Cleve included various minerals, and was in part already worked out by him. Results showed that Demarçay's crystallisation process with concentrated nitric acid is very well adapted to the separation of gadolinium in the case of a mixture of gadolinium and samarium. In order to discover how a mixture of gadolinium and yttrium would behave under similar circumstances, I made three crystallisations of such a mixture with atomic weight R = 143·4; the concentrated nitric acid used having the specific gravity 1·54.

The atomic weight of the earth in the first mother-liquor was determined, as also that of the crystals from the third mother-liquor, and the following values found:—

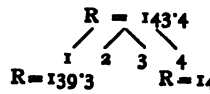


In the above scheme, 1, 2, 3 are the first, second, and third mother-liquors, and 4 the crystals from 3. From this result it is evident that the nitric acid crystallisation is a specially active means of separating gadolinium also from yttrium.

I have convinced myself by special tests that the nitrates of triatomic cerium, praseodymium, neodymium, and lanthanum do not crystallise, or only with difficulty, in concentrated nitric acid; hence gadolinium nitrate is less soluble in concentrated nitric acid than the nitrates of the remaining earths.

In one material, which was obtained as the first ammonia precipitates of a gadolinium-yttrium mixture, a holmium (or dysprosium) line was still visible after about fifteen re-crystallisations: this indicates that the nitrate of the element in question is difficultly soluble, as is that of gadolinium.

Successful separation of gadolinium from terbium was not attained by this means, for the gadolinium oxide obtained after several re-crystallisations is only slightly lighter in colour than the original material. I also carried out a fractionating experiment with the chlorides in concentrated hydrochloric acid, but with a much less satisfactory result, viz., with the same material as before the result thus obtained was—



The quickest method for obtaining pure gadolinium seems to me to be that which I used later, viz., to fractionate the nitrate in concentrated nitric acid after the weaker bases have been removed, and then partial precipitation with cold dilute ammonia. (The ammonia should always be used dilute, so that precipitation takes place slowly). By this means the terbium and samarium earths are obtained in the first precipitates; in the last, a faintly coloured and finally almost pure white gadolinium oxide is obtained. The traces of didymium which were still present after crystallisation of the nitrates are best re-

moved by a new series of crystallisations of the nitrates. The advantage of the crystallisation process is obvious, for by this means a greater number of operations can be carried out in the same time as a single tedious potassium-sulphate precipitation.

The concentrated nitric acid forms fairly easily supersaturated solutions of the nitrates, wherefore it is advantageous to add a few crystals to the solution.

During the progress of my work a quantity of nearly pure gadolinium earth was added to the material I had purified, by the kindness of Prof. Cleve.

Atomic Weight of Gadolinium.

As already mentioned, Marignac found for the equivalent weight of $Y_2\overset{II}{R}O = 120{\cdot}5$, which indicated an atomic weight of $\overset{III}{R} = 156{\cdot}75$ for gadolinium. Lecoq de Boisbaudran obtained from Marignac's original material, after further purification, the number 155 95, or, after allowing for the probable effect of impurities, 156·15. Cleve found 155 with his earth, in which Lecoq found later some samarium to be present, which had somewhat lowered the atomic weight. Bettendorff, whose determinations may be of greater value than the former, having been carried out with purer material, obtained as a mean result the number 156·33. It seems to me to have been proved that the atomic weight of gadolinium cannot be far from the number 156.

My own determinations, carried out in the usual way, by converting a weighed quantity of oxide into sulphate, give 156·38 as a mean result. But the fractional part of this value seems uncertain, and in the following I shall use the round number 156 to simplify matters. My object is to undertake a determination of the atomic weight later.

The remaining atomic weights used in my work are those of the Commission of the German Chemical Society, 1898.

(To be continued).

SEPARATION OF COPPER FROM CADMIUM.

By G. BORNEMANN.

Copper is precipitated quantitatively from its neutral boiling solutions by oxalic acid, even if we add four times the theoretical quantity of this reagent necessary; but the precipitate does not deposit well, and is difficult to wash, as it passes through the pores of the filter-paper. If the solution is made slightly acid with nitric acid we can get a much more rapid deposition of the precipitate, and the filtration is made much easier; but in this case, care must be taken not to use a large excess of oxalic acid, and a special paper must be used for filtration; the precipitate must be washed with water containing a little oxalic acid. The analysis of the oxalate of copper, dried at 110° till the weight is constant, leads to the formula $6CuC_2O_4 + H_2O$.

As for the separation of copper and cadmium by means of oxalic acid, it has been found that this separation is only possible in boiling nitric solution, otherwise a part of the cadmium is precipitated in the state of oxalate. Further, a small quantity of this salt separates if the nitric solution is cooled. The quantity of oxalic acid used should not exceed the quantity theoretically equivalent to the sum of the copper and cadmium present in the solution. On the other hand, a quantity of nitric acid equal to the quantity of oxalic acid may be added. The presence of ammoniacal compounds does not interfere with the precipitation.

The method of working is as follows:—

The moderately concentrated solution is made slightly acid with nitric acid, and then heated to boiling point and a boiling solution of oxalic acid (saturated in the cold) added, in quantity slightly greater than that of the copper present. The mixture is placed on a boiling water-bath, and left to stand until the supernatant liquid is perfectly clear. The liquid is then carefully decanted on to a filter, after the addition of a little nitric and oxalic acids, and the precipitate washed with acidulated water, and finally with boiling distilled water.

The precipitate of oxalate of copper is dried, detached as much as possible from the filter-paper, and placed in a Rose's crucible. The filter paper is burnt and the ash added to the bulk of the precipitate, a little sulphur is added, and the whole calcined in a current of hydrogen. Finally, the cuprous sulphate is weighed.

If the precipitated oxalate contains cadmium, a yellow stain of sulphide of cadmium will be observed on the crucible and on the surface of the cuprous sulphide.

The cadmium is determined in the filtrate in the usual manner.

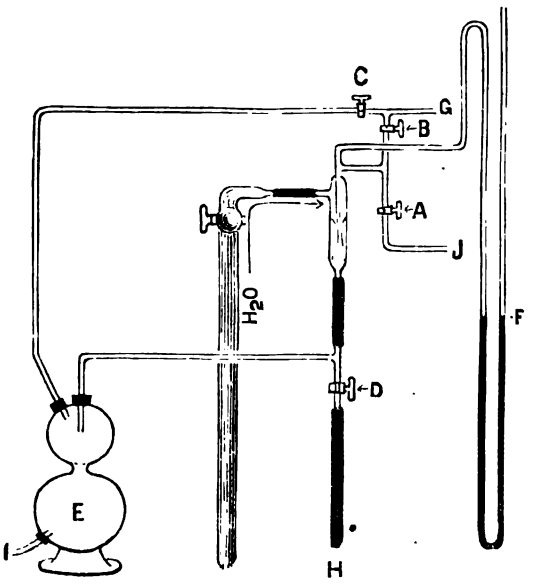
The author prepared a solution containing 6·200 grms. of CuO, and 9·885 grms. of CdO per 100 c.c.. On proceeding as above described he found an average of 6·210 grms. of CuO, and 9·872 grms. of CdO.—*Chemiker Zeitung*, 1899, xxiii. [53], p. 565.

A COMBINED EXHAUSTER AND BLOWER.

By EDWIN DOWZARD, F.C.S.

In the majority of chemical laboratories connected with manufacturing concerns, the supply of apparatus is not prodigal, and a chemist has frequently to utilise old and damaged pieces of apparatus.

In the accompanying illustration it is shown how, with the aid of a damaged Kipp's H_2S apparatus, an ordinary filter-pump may be converted into a combined exhauster and blower.



We will suppose that the water is turned on, and the taps A, B, C and D are open. If D and B are closed, the mixture of water and air from the pump will pass into the Kipp's apparatus, instead of passing through H; in a few seconds the outlet I will be covered, and a current of air will be delivered at G; of course the taps A and C must be left open. It is necessary to regulate the size of the outlet I, so that the water in E never reaches higher than the dotted line.

If we now open D and B, and close C, there will be suction at G and J; the vacuum so produced (if used to

exhaust distillatory apparatus, &c.) is measured by the gauge F.

Of course the taps A, B, C, and D may be replaced by burette clips, &c.

THE VOLUMETRIC ESTIMATION OF SULPHUR IN CAST-IRON, STEEL, &c.

By J. THILL.

By attacking the metal with hydrochloric acid in the well-known apparatus used for this purpose, the sulphur is liberated as sulphuretted hydrogen. The gas is received in 25 c.c. of a decinormal solution of arsenious acid, to which has been added 50 c.c. of a cold saturated solution of bicarbonate of soda. Care should be taken that the gas is not given off too rapidly.

When the attack is finished, the gas which still fills the apparatus is driven out by means of a current of carbonic acid, and the passage of this current of gas is continued until the hydrochloric acid carried with it has neutralised the alkaline solution, and precipitated almost the whole of the trisulphide of arsenic. This operation takes eight or ten minutes.

We then add a few c.c. of hydrochloric acid, make up the volume to 500 c.c., and filter. The filtrate is collected in a dry beaker, and 100 c.c. are taken, and titrated with N/50 iodine, after adding starch and a sufficient quantity of carbonate of ammonia to render the solution alkaline.

From the number of c.c. of iodine used, we subtract the number of c.c. required by 25 c.c of a decinormal solution of arsenious acid. The difference corresponds to the sulphuretted hydrogen given off. This result multiplied by 0·0024045, gives the amount of sulphur contained in the sample.—*Zeits. Anal. Chem.*, xxxviii., p. 342.

ELECTROLYTIC ESTIMATION OF ZINC.

By H. PAWECK.

For the electrolytic estimation of zinc the author has adopted the following method :—

The cathode is formed of one or two discs of sheet copper, 6 c.m. in diameter, suspended by a copper wire 10 c.m. long and 1 m.m. diameter attached to the centre; when two discs are used they should be about 12 m.m. apart. The discs should be 0·5 m.m. in thickness. Before fixing the disc or discs to the wire they should be cleaned by rubbing them with wet chalk; they should then be washed with water and plunged for an instant in dilute sulphuric acid.

The cathode thus cleaned is amalgamated electrolytically once for all, by suspending it in a bath containing about 0·6 grm. of bichloride of mercury and 3 to 5 c.c. of concentrated nitric acid per 200 c.c. In one hour, with a current of 0·1 to 0·2 ampère, a sufficient deposit of mercury is obtained on the cathode. The electrode thus amalgamated is washed with dilute hydrochloric acid, then with water, alcohol, and ether, and finally dried and placed in an exsiccator.

The anode is formed of a perforated strip of platinum.

The electrolytic estimation can be made either in alkaline or acid solution. The author gives the results of a large number of analyses to show the value of the method. In these analyses the electrolyte, as a rule, has a volume of 200 c.c., and contains—

Alkaline Electrolyte.

0·16—0·51 grm. of Zn, as $ZnSO_4$.
7—12 grms. of Seignette salt.
5—8 grms. of KOH or NaOH.

Acid Electrolyte.

0·23—0·31 grm. Zn, as $ZnSO_4$.
14 grms. K_2SO_4 or Na_2SO_4.
3—5 drops of concentrated H_2SO_4.

The electromotive force employed is 3·6 volts. When the electrolysis is finished, the cathode is washed in distilled water, alcohol, and ether, dried at a low temperature, and weighed. The deposit of zinc can be easily redissolved in hydrochloric acid of medium strength.—*Zeit. f. Elektrochemie*, v., [18], 221.

ON BINOXIDE OF MOLYBDENUM.

By MARCEL GUICHARD.

It has been shown (*Comptes Rendus*, cxxv., pp. 25 and 105; *Bull. Soc. Chim.*, Series 3, xvii., p. 902), that when trioxide of molybdenum or molybdic anhydride is gradually reduced by means of hydrogen, no other oxide than the brown anhydrous binoxide is formed. Particularly it is noticed that, at no matter what temperature, reduction does not lead to the formation of definite oxides, such as Mo_5O_{12}, Mo_2O_5, Mo_3O_8, or $3MoO_2 2Mo_3$, MoO_2MoO_3, Mo_2MoO_3. Several authors having described these compounds of the binoxide and the trioxide, that is to say, oxides intermediate between MoO_2 and MoO_3, we have taken up the study of their preparation.

I. *Action of Molybdic Anhydride on Molybdate of Ammonia.*

When we heat a well-powdered mixture of 1 part of molybdate of ammonia and 2 parts of pure molybdic anhydride, for a few minutes before the blowpipe in a covered platinum crucible, until the mass is fused, we obtain a brownish-violet button with red, violet, and copper coloured iridescence; if the heating has not been too prolonged the surface of the button is covered with a grey metallic layer and flakes of sublimed molybdic anhydride; there is also a little sublimate on the cover of the crucible.

The experiment, repeated a large number of times, always leads to the same result.

Berlin (*Journ. f. Prakt. Chem.*, xlix, p. 444), thought that a molybdate of molybdic oxide was thus produced; to get rid of the excess of the molybdic oxide which enveloped it, he treated it with ammonia; he then obtained the compound Mo_3O_8.

Uhrlaub (*Poggend. Ann.*, ci., p. 605), having shown that this oxide contains nitrogen, Muthmann (*Liebig's Ann.*, ccxxxviii., p. 117), followed the treatment with ammonia, by a prolonged boiling with hydrochloric acid, to destroy the nitrated compound. He thus obtained an oxide with the composition Mo_5O_{12}, not attacked by alkaline solutions or dilute sulphuric and hydrochloric acids.

When we attempted, by this method, to dissolve the molybdic anhydride in ammonia we found that the operation was a very long one. In one experiment, the body resulting from the action of molybdic anhydride on molybdate of ammonia, washed several times with ammonia by decantation, and then dried at 110°, contained 72·59 per cent of the metal. After being treated for several days with ammonia, it contained 74·39 per cent of molybdenum, and was not then completely freed from free molybdic anhydride. It is thus very difficult to effect a complete purification by means of ammonia. This is caused by the fact, that though molybdic anhydride dissolves very rapidly in ammonia, when it has only been heated to a dull red heat, it takes a very long time to dissolve after it has been melted or sublimed. On the other hand, a solution of soda dissolves molybdic anhydride, after calcination, fusion, or sublimation, with very great rapidity.

We have therefore adopted the following treatment :—

The oxide to be purified is treated on the water-bath alternately with a solution of soda at 10 per cent; pure hydrochloric acid diluted with its own volume of water is then added until the hydrochloric acid is no longer coloured red, and the liquid, saturated with acetic acid, is no longer precipitated by acetate of lead.

The oxide thus obtained, dried at 110°, is of a brownish-violet colour and crystalline; the iridescence apparent before the purification has disappeared; it was due to a thin layer of melted molybdic anhydride enveloping each particle of oxide. Under the microscope it appears in the form of small crystals with numerous brilliant facets. It does not contain any nitrogen, and gives on analysis:—Molybdenum, 74·90 and 75·01 per cent; calculated for MoO_2, 75 per cent. This oxide is therefore pure, crystallised binoxide of molybdenum.

The binoxide prepared by Bucholz's method, by heating the molybdate of ammonia alone, and then submitting it to the treatment as above described, has the same appearance, the same colour, and the same composition. On analysis it gave:—Molybdenum, 75·05 per cent.

II. *Electrolysis of Melted Molybdic Anhydride.*

Buff (*Liebig's Ann.*, cx., p. 275), in 1859, electrolysed melted molybdic anhydride in a U tube with platinum electrodes. The lower oxide produced, washed with ammonia, when analysed by Wöhler, had the composition Mo_3O_8.

According to Muthmann, this body is not formed by the electrolysis of the pure oxide MoO_3, and that obtained by Buff should contain nitrogen.

We have effected the electrolysis of molybdic anhydride in the following manner:—

The pure oxide, MoO_3, is melted in a porcelain crucible covered with a lid of asbestos card; two electrodes of graphite, 8 m.m. in diameter, pass through this cover and conduct the current from five Bunsen cells through the melted mass. The electrolysis is continued for thirty minutes with a current of 10 ampères. The difference of potential between the electrodes is about 3 volts.

The crucible contains, after cooling, a brilliant grey-brown iridescent button, under a layer of sublimed oxide, MoO_3. The electrodes do not show a trace of alteration. An experiment made with platinum electrodes gave the same result.

To prevent an abundant sublimation of molybdic anhydride, it is preferable to form the bath with molybdate of potassium, which gives the same results as molybdic anhydride alone.

The most convenient bath is obtained by the fusion of 15 grms. of the pure oxide, MoO_3, with 20 grms. of carbonate of potassium; the molybdate formed corresponds as nearly as possible to the quintamolybdate, $5MoO_3K_2O$, and gives off but very little vapour of molybdic anhydride when melted at a red heat.

Under these conditions, we were able to prolong the electrolysis by an hour and a half, with a current of 3·5 ampères. The return is always small, for the reason that, as observed by Buff, the oxide formed being a good conductor of electricity, a moment arrives when the current passes without producing any sensible electrolysis; the disengagement of oxygen at the positive electrode then becomes almost nil.

The lower oxide formed, mixed with a large excess of MoO_3, or melted molybdate of potassium, is treated with soda at 10 per cent until nothing more is dissolved; it is then treated with hydrochloric acid, water, and finally dried at 110°. There then remains a brilliant brownish-violet body, formed of an agglomeration of a large number of crystals. Analysis gives:—

	Electrolysis of molybdic anhydride	Electrolysis of molybdate of potassium.	Calculated for MoO_2.
	Per cent.	Per cent.	Per cent.
Molybdenum.. ..	74·95	74·83	75·00

This oxide is thus pure crystallised binoxide of molybdenum.*

The analysis of different samples of binoxide obtained by us, were made by two different methods:—

1. By oxidation of the oxide by means of dilute nitric acid, then desiccation and calcination below a dull red heat of the trioxide formed.

2. By the oxidation of the binoxide by means of dilute nitric acid and saturation of the molybdic acid formed by ammonia; the solution is then acidulated with acetic acid and precipitated with acetate of lead. The molybdate of lead dried, and moderately calcined, is weighed (Chatard's method).

These two methods are very exact. To sum up, the oxide produced by the calcination of a mixture of molybdate of ammonia and molybdic anhydride, is crystallised binoxide of molybdenum identical with that formed by the calcination of molybdate of ammonia alone.

On the other hand, the electrolysis of melted molybdic anhydride and molybdates of potassium also give the crystallised binoxide.

The result of this research shows that there is no other intermediate anhydrous molybdic oxide between the oxides MoO_2 and MoO_3 than the one produced by the dehydration of the blue hydrated oxide, if this dehydration can be effected without peroxidation.

We are about to examine this matter further.—*Bull. Soc. Chim.*, Series 3, xxi., No. 24.

* We shall, later on, give the results obtained by the electrolysis of other compounds of molybdenum.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 18th, 1899.

Professor Thorpe, F.R.S., President, in the Chair.

Messrs. E. W. Lewis, A. J. Shelton, S. Blofield, and J. N. Goldsmith were formally admitted Fellows of the Society

Certificates were read for the first time in favour of Messrs. Frederick Nolan Baker, 3rd Royal Inniskilling Fusiliers, Mullingar, Ireland; William Francis Cooper, Ashlyns Hall, Berkhampstead; Alexander Findlay, Emilienstrasse, 10[1], Leipzig, Germany; Julius Geldard, 120, Park View Terrace, Otley Road, Bradford; Adam Houston, Brisbane House, Bellahouston, Glasgow; Walter A. Riley, Brunswick Lodge, Newmarket Road, Norwich; Fred Pilkington Sargeant, Springfield Place, Leeds; Edward Shrapnell Smith, 35, Botanic Road, Wavertee Park, Liverpool, E.

Of the following papers, those marked * were read:—

*1. "*Note on Nitrogen Halogen Compounds.*" By Julius Stieglitz and E. E. Slosson.

The authors point out that F. D. Chattaway and K. J. P. Orton, in their work on "A Series of Substituted Nitrogen Chlorides, &c." (*Trans.*, 1899, lxxv., 1046), have overlooked a series of papers emanating from the Kent Chemical Laboratory of the University of Chicago, in which, since 1893, results of work on the nitrogen halogen compounds in various directions have been given, and a definite line of work undertaken for further investigation. In one of these papers "Ueber die Einwirkung von unterbromiger Saeure auf Saeureanilide" (*Ber.*, 1895, xxviii., 3265), the preparation and behaviour of acyl-phenyl nitrogen chlorides and bromides are discussed, and some of the compounds obtained by Chattaway and Orson described. In order to avoid future conflict, the line of work pursued by one of the authors and his students is fully outlined as centreing in a study of the "Beckmann

rearrangement" of acyl-nitrogen halides and analogous bodies.

Further results are given concerning the behaviour of acyl-phenyl nitrogen halides, especially towards alkalis, potassium cyanide, and zinc ethide. All of these reagents reproduce acylanilides. The preparation of acyl-alkyl-nitrogen chlorides and representatives of the series are described: *ethyl benzoyl nitrogen chloride*, $C_2H_5NCl.COC_6H_5$, m.p. 53·5°, the corresponding methyl compound, is an oil. Alkalis, potassium cyanide, and zinc ethide regenerate the acyl-alkyl-amides.

Further representatives of the isomeric series, the chloro (or bromo) imido-acid esters, $RC(:NCl)OC_2H_5$, &c., are given; *chloroimidoethyl metanitrobenzoate*, $NO_2C_6H_4C(NCl)OC_2H_5$, m. p. 61°, the corresponding *bromide* melts at 71°; the analogous *chloroimide* of *β-naphthoic ethyl ether* melts at 68°, the *bromide* at 77°. These ethers are remarkably stable nitrogen halides, perhaps the most stable known. Some have been distilled without decomposition in a vacuum above 100; boiling alcoholic ammonia does not decompose chloroimidoethyl-metanitrobenzoate. Although a number have been obtained in solid form, no stereoisomeric compounds, $RC(:NCl)OR$ have as yet been observed, such as exist in the case of the analogous benzhydroximic ethers, $C_6H_5C(:NOH)OC_2H_5$. On treatment with zinc ethide, ethylamine derivatives are obtained.

Discussion.

Dr. Chattaway said he regretted that the papers mentioned had been at first overlooked. Dr. Orton and he had approached the subject from an entirely different point of view, and he did not think that their future work was likely to overlap. Professor Stieglitz and his students were working on the Beckmann change, while they were chiefly studying substitution, and endeavouring to show that the chlorination and bromination of anilines and anilides are not direct processes, but that nitrogen chlorides and bromides are always first formed, and under favourable circumstances subsequently undergo isomeric change.

*2. "*On the Electrolysis of the Nitrogen Hydrides and of Hydroxylamine.*" By E. C. Szarvasy, Ph.D.

A short account was given of the experiments of the author on the electrolysis of solutions of ammonia, hydrazine, azoimide, hydroxylamine, and of their salts.

The experiments were carried out at different temperatures, and with varying current densities and concentrations. The conditions were determined under which ammonia, hydrazine, and azoimide, on electrolysis, yield their two components in the proportions contained in their molecules respectively, and without the formation of any secondary products.

In the case of hydroxylamine and its salts, both oxidation and reduction products were formed, no matter how the experimental conditions were varied.

Many attempts were made to prepare polymeric nitrogen by electrolysing solutions of azoimide and its salts at very high current densities; this part of the work is still in hand.

*3. "*On the Relationship between the Constitution of some Substances and the Fluorescence which they exhibit.*" By J. T. Hewitt, M.A., D.Sc.

Since fluorescence is the absorption of radiant energy of definite wave-length or wave-lengths, and emission of the same energy after it has been reduced in vibration-frequency, one would expect to find fluorescence usually a property of substances capable of existing in tautomeric forms. As a matter of fact, the colouring-matters which do exhibit fluorescence are almost without exception tautomeric, the exceptions which are known being usually substances for which no very great degree of purity can be guaranteed.

But many substances which behave tautomerically do not exhibit well-marked fluorescence; this might well be due to the fact that in a solution in which the two tautomeric forms are in equilibrium the reaction velocities from one form to the other are so small that a very few of the molecules have the opportunity of absorbing energy when they correspond to one and emitting it in the other configuration.

The case is, however, very different when the molecule in one of its configurations is symmetrical; to pass from this to the other configuration, either of two displacements is necessary, equal in magnitude, but opposite in direction. Just as a pendulum (neglecting the friction due to its support and the medium in which it vibrates) swings regularly between two extreme positions, passing at regular intervals through its lowest point, so a molecule of a substance which exhibits the kind of tautomerism characteristic of fluorescein might undergo continual and frequent change. Fluorescein itself yields derivatives, not only of a phenolic lactone, but also of a quinone-carboxylic acid. The transition from the lactone to the quinone-carboxylic form may be effected by displacement of either of its phenolic hydrogen atoms, the symmetry of the molecule conditioning the equality of energy necessary for displacement of either atom. Hence all the molecules will be undergoing tautomeric change continuously and frequently, and energy absorbed when the molecules have one configuration will be, to an appreciable extent, emitted when they correspond to the other configuration. It is practically certain that the vibration frequency of fluorescein is different in the two states (the diethyl ethers corresponding to the two forms are quite different in the character of their absorption), and hence every opportunity is offered for energy of a rapid vibration frequency to be largely transformed into energy of greater wave-length.

In the case of fluorescein, the condition of a molecule may be symbolised in the manner shown below.

The greater number of fluorescent dye-stuffs conform to the theory shortly stated above; exceptions may, however, be classed under two heads:—

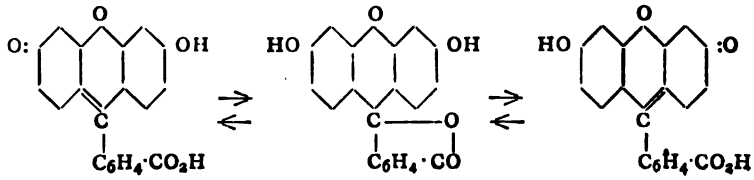
(i.) Substances having the requisite structure, but not exhibiting fluorescence.

(ii.) Substances exhibiting fluorescence, but not possessing the doubly symmetrical tautomeric structure considered in the case of fluorescein.

It is, however, probable, that many of the exceptions are only apparent, the fluorescent spectrum not having been sufficiently examined.

4. "*Action of Fuming Nitric Acid on α-Dibromocamphor.*" By Arthur Lapworth and Edgar M. Chapman.

The authors have repeated the work of Kachler and Spitzer on the oxidation of α-dibromocamphor (*Monatsh.*, 1883, iv., 554), and agree with them that camphoronic acid is produced, but the quantity of this substance is small, and it is difficult to separate it from homocamphoronic acid, which is present in much larger amount; on the other hand, they have not been able to detect any isocamphoronic acid, and conclude that Kachler and



Spitzer were merely dealing with impure homocamphoronic acid.

The product, to which Kachler and Spitzer gave the provisional formula $C_{24}H_{33}BrN_4O_{12}$, appears to have been a mixture of the substance $C_{10}H_{16}N_2O_6$, already described by the authors (*Trans.*, 1899, lxxv., 986), with ordinary nitrobromocamphor, $C_{10}H_{14}OBrNO_2$, produced by the nitration of monobromocamphor formed in the initial stage of the reaction.

Besides the above-mentioned products and dibromocampholid, a number of other substances seem to be formed. One of these remains dissolved in the water with which the mixture is diluted, and cannot be isolated by dilution. It is obtained by extracting the liquid containing the homocamphoronic acid with chloroform, and forms a neutral oil which is not readily soluble in pure water; it is probably a lactone. When this substance is allowed to remain with aqueous potash, it produces an acid having the formula $C_{10}H_{16}O_4$, which forms magnificent transparent crystals, is sparingly soluble in water, and melts at 177°. When distilled, or warmed with strong acids, it undergoes a curious molecular change, yielding an acid, $C_{10}H_{12}O_2$, melting at 167—168°, which has been identified as trimethylbenzoic acid, (CO_2H : Me : Me : Me :: 1 : 2 : 3 : 4).

5. "*Note on Volhard's Method for the Assay of Silver Bullion.*" By T. K. Rose, D.Sc.

The precautions to be observed in using the method are described, and the limit of accuracy put at 0·1 per 1000, instead of 0·25 per 1000, as stated by Van Riemsdyk.

6. "c-*Substituted Hydroxytriazoles.*" By George Young, Ph.D., and Ernest Witham, B.A., B.Sc.

The authors have prepared hydroxytriazoles by two actions represented by the equations—

$$R\cdot CH{:}N\cdot NH\cdot CO\cdot NH_2 + O = R\cdot C\begin{matrix}=N\cdot NH\\ \diagdown\!\!\diagup N\end{matrix}C\cdot OH + H_2O,$$

$$R\cdot CHO + NH_2\cdot CO\cdot N{:}N\cdot CO\cdot NH_2 =$$
$$= R\cdot C\begin{matrix}=HN\cdot N\\ \diagdown\!\!\diagup N\end{matrix}C\cdot OH + NH_3 + CO_2.$$

Where R is the same group, the products of the two actions are apparently identical.

c-*Phenylhydroxytriazole*, $C_6H_5\cdot C_2N_3H_2O$, m. p. 321—322°. Forms two silver salts, $C_6H_5\cdot C_2N_3HO\cdot Ag$ and $C_6H_5\cdot C_2N_3O\cdot Ag_2$. Acetyl derivative,—

$$C_6H_5\cdot C_2N_3HO\cdot C_2H_3O,$$

m. p. 248°. c-*Metanitrophenylhydroxytriazole*,—

$$NO_2\cdot C_6H_4\cdot C_2N_3H_2O,$$

m. p. 304. Forms two silver salts, $C_8H_5N_4O_3Ag$ and $C_8H_4N_4O_3Ag_2$. Acetyl derivative, $C_8H_5N_4O_3\cdot C_2H_3O$, m. p. 261—262°. *Cinnamalsemicarbazone*,—

$$C_6H_5\cdot CH{:}CH\cdot CH{:}N\cdot NH\cdot CO\cdot NH_2,$$

m. p. 215—216°. c-*Styrenylhydroxytriazole*,—

$$C_6H_5\cdot CH{:}CH\cdot C_2N_3H_2O,$$

m. p. 311°. Forms two silver salts, $C_{10}H_8N_3OAg$ and $C_{10}H_7N_3OAg_2$. Monoacetyl derivative, $C_{10}H_8N_3O\cdot C_2H_3O$, m.p. 241—242°. Diacetyl derivative, $C_{10}H_7N_3O\cdot(C_2H_3O_2)$, m.p. 137—138°.

7. "*Note on the Use of a Mixture of Dry Silver Oxide and Alkyl Halides as an Alkylating Agent.*" By G. Druce Lander.

Dry silver oxide and alkyl iodides have been employed in the preparation of the ethereal salts of alkyloxy-acids (*Trans.*, 1899, lxxv., 157, 485, 754). The use of the method in the preparation of alkyl derivatives of compounds of other classes has been studied. Optically active ethyl menthyl ether has been prepared by the action of dry silver oxide and ethyl iodide on *l*-menthol. The optically active ether is an oil boiling at 207·5—209·5° (uncorrected); $d20°/4° = 0·8537$: $[\alpha]_D\ 20° = -98·32°$.

The ethyl and isopropyl ethers of benzoin are obtained by the action of silver oxide and ethyl and isopropyl iodides respectively on benzoin. The ethyl ether crystallises from light petroleum in needles melting at 58—58·5°; compare Limpricht and Jena (*Annalen*, 1870, clv., 96), and Fischer (*Ber.*, 1893, xxvi., 2415). The isopropyl ether, of similar crystalline form, melts at 72—75°.

The action of silver oxide and ethyl iodide on benzamide leads to the formation of ethyl benzimido ether. The hydrochloride melts at 119—120° with evolution of gas; compare Pinner (*Ber.*, 1883, xvi., 1654), and Tafel and Enoch (*Ber.*, 1890, xxviii., 103).

The introduction of one ethyl group in place of hydrogen in ethyl acetoacetate can be readily effected by the aid of silver oxide and ethyl iodide. The product consists of ethyl ethylacetoacetate containing, however, approximately 2 per cent of ethyl β-ethoxycrotonate, melting at 30°, and yielding on hydrolysis ethoxycrotonic acid which melts at 137·5°, with evolution of gas. The introduction of an ethyl group into ethyl ethylacetoacetate, and ethyl malonate by the same method, is also possible, but the proportion of the ethyl homologue obtained is small. No indication could be obtained of the production of the ethyl homologue of ethoxycrotonic acid by the action of silver oxide and ethyl iodide.

Ethyl acetosuccinate may be prepared by the action of silver oxide and ethyl iodoacetate upon ethyl acetoacetate.

Further results and details will be given in a subsequent communication.

Victor Meyer Memorial Lecture.

The Victor Meyer Memorial Lecture will be delivered by Professor T. E. Thorpe, F.R.S., President of the Society, on the evening of Thursday, February 8th, 1900, at 8.30.

PHYSICAL SOCIETY.

Ordinary Meeting, January 26th, 1900.

Prof. Lodge, F.R.S., President, in the Chair.

A paper by Prof. Ayrton and Mr. Mather on "*Some Developments in the use of Price's Guard Wire in Insulation Tests*," was read by Prof. Ayrton.

For insulation tests made by the direct deflection method the guard-wire properly applied affords complete protection against surface leakage when the ends of the cable tested are near the galvanometer, so that it is possible to have the wire connecting the conductor of the cable with the galvanometer terminal "air insulated." A difficulty, however, arises when the ends of the cable are at a considerable distance from the testing instrument; this may render air insulation impossible. The authors have overcome this difficulty by applying a guard-wire along the entire length of the lead. This is done by using a concentric wire to connect the cable and galvanometer, the inner of the concentric being used as the lead, and the outer as the guard-wire. The principle can also be applied to determine whether a defective piece of cable is bad throughout, or bad owing to one or more isolated faults. In this case the cable is placed in two water-tanks, one of which is earthed, and the other fairly well insulated. By a suitable arrangement of the guard-wire it is then easy to determine the resistance of the wire in the earthed tank, so that by altering the length of this wire the character of the insulation can be determined throughout the whole length of the cable. In referring to some of the earliest experiments with the guard-wire made by Mr. Appleyard in 1895, Prof. Ayrton pointed out that the principle had not been applied completely, and that at one point there was a chance of leakage.

Mr. Campbell said that the necessity of having a concentric could be obviated by simply hanging the lead from the guard-wire by short lengths of material of fair insulation.

Mr. Appleyard said that he quite agreed with Prof. Ayrton that the guard-wire ought in general to be applied at both ends of all leads, provided that both ends could be got at. The reason it was used at one end only in the experiments on dielectrics, made in 1895, was that the far end of the lead was carried into the condenser box, which was submerged in water in the temperature tank. Special precautions were taken to ensure good insulation of the submerged end of the lead, and tests showed that the leakage there was nil. As the end of the wire could not be got at, no guard-wire could be applied. Mr. Appleyard congratulated the authors upon the use of a concentric cable for a lead, and pointed out that such a lead was sufficient for all the routine tests on core; the inner and outer conductors could be used for the purpose of taking the "copper" resistance.

Mr. Price expressed his interest in the developments of his principle which had been made by the authors.

Mr. Appleyard then read a paper on "*A Fault-test for Braided and other Cable-core.*"

This method enables the fault to be found without the removal of braiding or tape. The core is wound on two insulated drums or tanks, the intermediate piece of cable being about ten feet long. One end of the core is left free, the other is connected to earth through a galvanometer and a battery. A guard-wire is connected from some point between the galvanometer and the battery to some point of the braiding on the wire between the drums. A wet cloth, connected to an earth wire, is laid on one or other of the drums, over the braiding. The galvanometer deflection is noted. The earth-wire is then changed over to the second drum, and the corresponding deflection is observed. A comparison of these deflections at once indicates upon which drum the fault lies. With the galvanometer still deflected the core may be run through a suitable contact brush or sponge attached to the guard-wire. The instant the fault passes under the guard-wire contact, the deflection falls and the fault is located. The paper gives the theory of the method, and indicates how to apply it (1) to localising "distributed" faults; (2) to several faults in a single cable; and (3) to the case of a single fault. One advantage of the method is that at the critical moment when the fault passes under the guard-wire the galvanometer is short-circuited through the fault and thus completely protected.

A paper on "*Reflection and Transmission of Electric Waves along Wires,*" by Dr. E. Barton and Mr. L. Lownds, was read by Dr. Barton.

The waves used were produced by means of an induction coil and an oscillator, and travelled along wires 0·15 c.m. diameter, 8 c.m. apart, and 166 metres long. The ends of the wires were connected by graphite markings on ground glass, so that any wave trains which reached the ends were at once absorbed. Three circular parallel-plate condensers were used of 15, 9, and 5 c.m. radius respectively. The plates were, in all cases, separated by air, and were placed 1 c.m. apart. The needle of the electrometer connecting the wires was uncharged, so that it was always attracted by the charged plates. The positions of the condenser and electrometer could be varied so as to study either the reflected or the transmitted waves. The electrometer produced a negligible disturbance, as it reflected only 0·04 per cent of the energy incident upon it. The authors have attacked the problem mathematically, using the relations of Heaviside, and have obtained expressions for the reflected and transmitted systems. These expressions consist of two terms, one of which is comparatively unimportant. From the other term certain values have been calculated. A superior limit has then been given to the other term, and the values already obtained have been subjected to a correction on this account. By a suitable arrangement of the condenser and electrometer these calculated values have been experimentally determined, and are in close agreement with the theoretical numbers, falling in many cases between the results derived from the approximate and the corrected theories. The authors have also investigated the stationary wave system produced by interference when the electrometer is placed close to the condenser, and between the condenser and the oscillator.

The Chairman said that the experiments afforded a satisfactory verification of Heaviside's theory.

A paper on "*The Frequency of Transverse Vibrations of a Stretched Indiarubber Cord,*" by Mr. T. J. Baker, was taken as read.

In this paper Mr. Baker has investigated the frequency of the note given out by an indiarubber cord of square section when subjected to different tensions. The relation between length and tension is linear over a considerable range. The curve connecting length with frequency shows that while the cord was doubling its length the pitch was rising rapidly, but that further extension was practically without effect. Since the relation between length and tension is linear, while the sectional area is decreasing, it follows that the value of Young's modulus must be changing. The author has shown that the value of Young's modulus is proportional to the square of the stretched length of the cord. Using this fact the frequency of the note given out by a stretched indiarubber cord is shown to be proportional to a quantity which varies very slightly with increase in length of the cord, and hence the variation of elasticity is given as the cause of the constancy of the note.

Mr. Appleyard exhibited some Mirrors produced inside incandescent electric lamps by the application of voltages much above those for which the lamps were designed, and the consequent deflagration of the filaments.

The meeting then adjourned until February 9th.

NOTICES OF BOOKS.

The Analysis of Food and Drugs. Part II. *The Chemical and Biological Analysis of Water.* By T. H. Pearmain and C. G. Moor, M.A. London: Baillière, Tindall, and Cox. 1899. Pp. 172.

Natural waters, generally speaking, may be arranged in the following order with regard to purity:—(1) Rain-water collected under proper conditions; (2) Deep spring and artesian-well water; (3) upland surface waters; (4) subsoil water; (5) rivers water; (6) land springs; and (7) surface water from cultivated lands. The impurities found in water which affect its potability are mainly the products of decomposition of animal and vegetable matter, and also suspended matter, either living or dead, in the form of algæ, moulds, bacteria, insects, &c. Of late years less importance has been given to merely chemical results in the examination of water, while the value and importance of biological and bacterial examination cannot be over estimated. A water must not be condemned simply because it contains a high proportion of organic matter, if the bacteriological results show that only a small number of harmless microbes are present; and it must be further remembered that there may be distinct danger in carrying the bacterial purification of water too far; certain microbes act as policemen, so to speak, and are able to destroy pathogenic microbes introduced in small numbers. If, however, the police microbes become too few in number, the injurious ones increase at an enormous rate, and danger of an epidemic of disease is the immediate result.

The authors devote a considerable portion of their book to the chemical analysis of water, many of the various methods in vogue being well and clearly described, though we are sorry to see that very little space is devoted to Frankland's combustion process, which is unrivalled for the estimation of the organic carbon and nitrogen in water,

The bacteriological examination of waters is also soundly dealt with, the authors being known as authorities on this subject.

Altogether this book, dealing as it does with both the chemical and bacteriological branches of the subject, combines in one volume an amount of information that cannot fail to be of much service to those interested in the subject.

The Production of Tin. By Henry Louis, M.A., A.R.S.M., &c., Professor of Mining, Durham College of Science. London: *The Mining Journal.* Pp. 39.

The articles forming this volume were originally written for *The Mining Journal*, but are now presented in book form. They give a history of the production of tin from early times up to the present day.

Tin is by far the rarest of the common metals, and is produced in much smaller quantities than any of the others; it is but sparsely distributed throughout the world, and is only found in paying quantities in a few localities, such as Cornwall, Saxony, Spain, Bolivia, the Malay Peninsular, and Australia. It may be noted that tin is the only common metal besides iron, the only true ore of which is the oxide; it is, therefore, easily treated for producing the metal. Tin is reduced either in the blast-furnace or in the reverberatory furnace; the former is, however, probably responsible for 60,000 out of the world's approximate total of 80,000 tons. The relative importance in production of the different tin-bearing districts is shown by the following figures:—

Straits Settlements	60·6 per cent
Netherlands, East Indies	19·0 ,,
Australasia	7·9 ,,
Cornwall..	6·1 ,,
Bolivia	6·0 ,,
Other minor producers..	0·4 ,,
	100·0

It is to be hoped that before long a payable method of recovering tin from scraps and cuttings of tin-plate will be discovered. Such a process would materially help to diminish the price of the metal. There is also room for improvement in the methods in use for producing tin, in fact it is shown that there has been less progress in the metallurgy of tin than in that of any other metal; the best hopes for the future must, therefore, lie in the successful application of modern science to all branches of the industry, from mining to refining.

CORRESPONDENCE

PHYSICAL EQUILIBRIUM OF MOLECULAR SYSTEMS.

To the Editor of the Chemical News.

Sir,—In my letter to last week's Chemical News I suggested an explanation of the "Double Centre of Stability" of Si_2Cl_6. My friend Mr. T. Beacall, however, has called my attention to the fact that if there was present an *impurity* in the Si_2Cl_6, it may happen that this impurity would react with the Si_2Cl_6 *between the limits* 350—800°, and thus cause its dissociation, much in the same way that H_2O dissociates NH_4Cl vapour. Above 800° C. this *impurity* might itself be decomposed by the heat, and would thus cease to decompose the Si_2Cl_6. This would explain the stability of the Si_2Cl_6 above 800°.

It would therefore be advisable to repeat the experiments with *absolutely pure* Si_2Cl_6, and see whether an analogous phenomenon occurs with the *pure* product.—I am, &c.,

Geoffrey Martin.

13, Hampton Road, Bristol.
January 22nd, 1900.

RELATIONS BETWEEN THE ATOMIC WEIGHTS AND PHYSICAL PROPERTIES OF ELEMENTS.

To the Editor of the Chemical News.

Sir,—In the Chemical News (lxxx., p. 282) there appears a note from Mr. Thomas Bayley, pointing out that the product of melting-point (absolute) × coefficient of expansion for 1° C. is a constant for a number of elements.

In a paper I read before the Physical Society in June, 1895 (Chem. News, lxxi., p. 303), I brought forward the same relation, limiting the constancy of the product to elements in the same periodic group: about the same time Pictet proposed a similar relation, but introduced into the equation the cube root of the atomic volume, and applied the relation generally to all the elements.

I published a second paper in the Chemical News (lxxvi., p. 81), wherein I attempted to collate and criticise work that had been done by myself and other workers on this and some other allied relations.—I am, &c.,

Noel Deerr.

26, South Parade, Chelsea, S.W.,
January 23rd, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 2, January 8, 1900.

Application of the Law of Phases to Alloys and Rocks.—H. Le Chatelier.—By applying Gibbs's law of phases to a mixture of solid bodies taken at ordinary temperatures, and obtained by a succession of reversible transformations, such as solidification by freezing or crystallisation from solution, the author arrives at the following conclusion:—The stable state of a solid mixture (melted salts, metallic alloys, rocks, &c.), corresponds to a monovariant system—that is to say, that the number of phases should be equal to the number of independent constituents which enter into its composition.

Rhodicyanides.—E. Leidié.—The author attempted to reproduce cyanide of rhodium and potassium, first by the dry method and then by the wet method, in order to verify Marius's work. He found that the double cyanide crystallises in monoclinic prisms, slightly tinted yellow. These are anhydrous and extremely soluble in water. The nitrogen was estimated by an elementary analysis, the potassium by transformation into the chloride (calcination with NH_4Cl in a current of hydrogen), and the rhodium by electrolysis (after decomposition of the salt with H_2SO_4), This analysis leads to the numbers $Rh : Cy_6 : K_3$, possibly $Rh_2Cy_{12}K_6$. This latter formula has been proved by other considerations.

New Micro-chemical Reactions of Copper.—M. Pozzi-Escot.—By using crystallised cupric iodide the author obtained ammoniacal compounds which are very characteristic. First, the iodide, $CuI_2.4NH_3.H_2O$, which appears under the microscope in the form of little blue tetrahedra, and which salt was obtained by treating a cuprous ammoniacal solution with ammonium or sodium iodide. Secondly, a dissociable compound, of which the exact composition has not been obtained, but which appears to have the formula $CuI_4.4NH_3$, which can be prepared by adding the cupric solution to a quantity of ammonia, sufficient to bring the temperature up to the point of dissolving the copper hydrate (at about 40°); iodide of sodium or ammonium is then added. Under these conditions the liquor immediately becomes yellowish green and deposits rhombohedral tables of a very dark brownish black.

Action of the Magnetic Field on Becquerel Rays. Rays Deviated and Rays Undeviated.—P. Curie.—The author shows that the rays of radium are composed of two well defined groups, those which are deviated by a magnetic field and those which are not. The non-deviable rays appear to be quite analogous to the rays of polonium.

Penetration of Becquerel Rays Non-deviable by the Magnetic Field.—Mdme. Sklowdowska Curie.—The non-deviable rays are much less penetrating than the deviable rays. A more complete study of the penetration of the two kinds of rays across various substances show that their nature is entirely different, and confirms the results obtained by the examination of the effect of the magnetic field.

MISCELLANEOUS

The Huddersfield Town Council at their meeting voted Mr. Edward A. Harman, M.Inst.C.E., the Gas Engineer, an increase of salary of £100 per annum from January 1st this year, with an additional £100 increase on January 1st, 1901.

Estimation of the Available Molybdenum in Molybdenite.—H. Bornträger.—One grm. of the mineral is digested for two hours with 25 c.c. of concentrated nitric acid in an Erlenmeyer flask. The molybdic acid formed is re-dissolved in ammonia, the solution is filtered, and the residue again treated with nitric acid and ammonia. The filtrates are added together, acidulated with nitric acid, and evaporated to dryness. The dry residue is digested with alcohol at 50 per cent, which dissolves the nitrate of ammonia, and leaves the molybdic acid intact. The latter is collected on a tared filter, and weighed. It may also be dissolved in normal ammonia and estimated by titration.—*Zeit. Anal. Chem.*, xxxvii., p. 438.

Estimation of Sulphur in the presence of Iron (Pyrites).—O. N. Heidenreich.—To prevent the carrying down of ferric sulphate by sulphate of barium precipitated in a solution rich in iron, the author proposes to first reduce the iron by means of metallic zinc before proceeding with the precipitation of the sulphuric acid. The ferrous sulphate thus formed is not carried down. The sample of pyrites to be examined is attacked in the ordinary manner with aqua regia, and the solution obtained is diluted to about 250 c.c., and a certain quantity of zinc added. After complete decolouration the solution is rapidly filtered and made up to 600 c.c.; it is then heated to boiling and precipitated with a boiling solution of chloride of barium.—*Zeitschr. Anorg. Chem.*, 1899, xx., p. 233.

Estimation of Zinc and Manganese in the State of Sulphides.—E. Murmann.—The estimation of zinc and manganese by precipitation as sulphides and calcination of the precipitates in a current of hydrogen gives good results, but requires very careful manipulation on account of the difficulty experienced in obtaining a clear filtrate, and also on account of the long time necessary for washing. If the precipitation is carried out in the presence of bichloride of mercury, the finely divided sulphide of zinc or manganese is carried down with the sulphide of mercury, and deposited very rapidly. The filtration and washing are also very rapid; if the precipitate is not very abundant the pores of the filter may be first filled up with a little sulphide of mercury. On calcining the sulphide of mercury is completely volatilised, and the residue consists of perfectly pure sulphide of zinc or manganese.—*Monatsh. für Chem.*, xix., [7], 404.

Patera's Method for the Estimation of Uranium.—H. Bornträger.—Patera's method for the estimation of uranium in its ores is as follows:—The mineral is dissolved in nitric acid, the solution is diluted, saturated with carbonate of soda, heated to boiling, and filtered. The uranium is precipitated in the clear liquid by means of caustic soda; the precipitate of uranate of soda is collected and weighed as such. According to the author, this method, although sufficiently exact in the case of pure minerals, gives results which are much too high, in the case of samples rich in silica, as the precipitate will then contain a considerable proportion of silicic acid. The precipitate must in such a case be treated with hydrochloric acid, evaporated to dryness, taken up with dilute hydrochloric acid, filtered, and the uranium in the clear liquid reprecipitated by means of caustic soda or ammonia.—*Zeit. Anal. Chem.*, xxxvii., p. 436.

MEETINGS FOR THE WEEK.

MONDAY, 5th.—Society of Arts, 8. (Cantor Lectures). "The Nature and Yield of Metalliferous Deposits," by Bennett H. Brough.
—— Society of Chemical Industry, 8. "On Recent Objections urged against the Adoption of the Metric System," by Dr. W. S. Squire, F.I.C., F.C.S. "Oil of Carthamus Tinctorius (Safflower Oil)," by H. R. Le Sueur.
—— Royal Institution, 5. General Monthly Meeting.
TUESDAY, 6th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
WEDNESDAY, 7th.—Society of Arts, 8. "Housing of the Poor," by Edmund Wilson.
—— Society of Public Analysts, 8. "Note on the Separation of Oleic Acid from other Fatty Acids," by J. Lewkowitsch, Ph.D., F.I.C. "Analysis of a Sample of 'Treacle' and of a Sample of so-called 'Golden Syrup,'" by C. G. Matthews, F.I.C., and A. Hyde Parker. "The Determination of Carbon and Sulphur in Steel," by Bertram Blount, F.I.C. "Note on Sour Milk," by H. Droop Richmond, F.I.C., and J. B. P. Harrison. "Butters from various Countries Compared," by C. Estcourt, F.I.C.
THURSDAY, 8th.—Royal Institution, 3. "Modern Astronomy," by Prof. H. H. Turner, M.A., F.R.S.
—— Society of Arts, 4.30. "New Projects of Railway Communication with India," by James Mackenzie Maclean, M.P. (This meeting will be held at the Imperial Institute).
—— Chemical, 8.30. Victor Meyer Memorial Lecture, by Prof. T. E. Thorpe, F.R.S., President.
FRIDAY, 9th.—Royal Institution, 9. "Symbiosis and Symbiotic Fermentation," by Prof. J. Reynolds Green, Sc.D., F.R.S.
—— Physical, 5. (Annual General Meeting). Address by the President, Prof. O. J. Lodge, D.Sc., F.R.S.
SATURDAY, 10th.—Royal Institution, 3. "The Idea of Tragedy in Ancient and in Modern Drama," by W. L. Courtney, M.A., LL.D.

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MICHAELMAS TERM.—Monday, October 2, to Saturday, December 16.
LENT TERM.—Monday, January 8, to Saturday, Apr 7.
EASTER TERM.—Monday, April 30, to Saturday, July 28.

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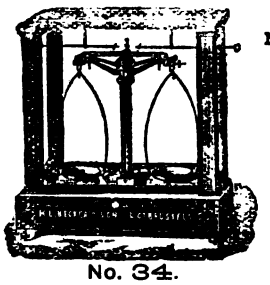

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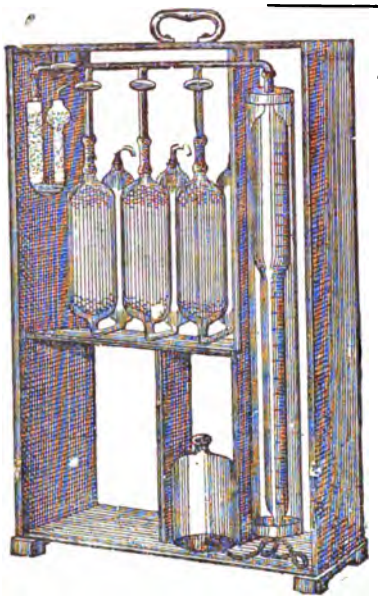

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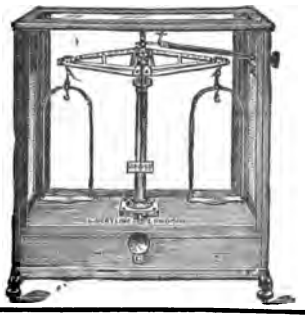

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THE CHEMICAL NEWS.

Vol. LXXXI., No. 2098.

ON THE BEHAVIOUR OF THE BECQUEREL AND RÖNTGEN RAYS IN A MAGNETIC FIELD.*

By the Hon. R. J. STRUTT, B.A., Scholar of Trinity College, Cambridge.

In the current number of *Wiedeman's Annalen* an experiment is described by Giesel showing that the Becquerel rays are deflected in a magnetic field. This result is of great interest, on account of the light which it throws on the nature of the rays. Up to the present, the evidence has tended to show that the Becquerel rays were of the same nature as the Röntgen rays, both being capable of penetrating thin metal sheets, of affecting a photographic plate, and of producing ionisation in the surrounding air. Neither could be refracted or reflected, and, so far has as yet appeared, neither could be polarised.

These facts seemed to form a fairly strong body of evidence that the two kinds of radiation were essentially similar. But the announcement of the magnetic deflectibility of the Becquerel rays seems to throw doubt on this conclusion. The Röntgen rays, so far as is known, are quite unaffected by magnetic force. Under these circumstances it seemed worth while to make a new attempt to discover such an effect on the Röntgen rays. This attempt I have carried out. It will be best to say at once that the result is negative.

A focus tube was employed as the source of radiation. It was placed at a distance of about 35 c.m. from a powerful electro-magnet, and in such a position that the cathode rays in the tube were parallel to the magnetic force due to the magnet. The line joining the oblique anti-cathode to the centre of the magnetic field lay in the plane of the anti-cathode.

A short distance in front of the magnet a wire was placed at right angles to the direction of the rays, and in the plane of the anti-cathode. It was thus at an angle of about 50° to the magnetic force—the same angle as that between the axis of the cathode stream and the anti-cathode. This wire was used to cast a shadow on a photographic plate placed at a distance of 65 c.m. on the other side of the magnet.

An exposure was first made with the magnetic force in one direction. The exposure was then stopped, the field reversed, and another exposure given, of course without shifting the plate. If then the rays had been appreciably deflected, the photograph should have shown two shadows, either overlapping or altogether separated.

The rays casting the shadow were those emitted at a grazing angle from the anti cathode. The reason for using these very oblique rays was that, owing to the foreshortening of the anticathode, the source was virtually narrower than it would have been had rays been used which left the anti-cathode at a greater angle. Thus sharper shadows were obtained, and a smaller magnetic deflection could have been detected.

The tube was arranged with its cathode stream parallel to the magnetic field, so as to avoid any shifting of the source of radiation when the magnet was reversed, owing to an effect of the magnet on the original cathode beam. Such a shifting would have given rise to a spurious effect. The only objection to this was that the shadow-casting wire had to be obliquely placed so as to be in the plane of the anti-cathode. Thus some sensitiveness was lost.

A Paper read before the Royal Society, January 18, 1900.

I shall now give an estimate of the smallest deflectibility which could have been detected. The rays traversed a distance of 65 c.m. after leaving the magnetic field.

It was estimated that a lateral displacement of the shadow of the wire by 0·02 c.m. could have been detected. But the wire was inclined 50° to the resultant magnetic force. Thus the smallest real displacement that could with certainty be detected was—

$$\frac{0{\cdot}02}{\sin 50} \text{ c.m.}$$

The smallest angular deflection of the rays which could be detected would be, in circular measure,—

$$\frac{0{\cdot}02}{65 \sin 50} = 0{\cdot}000405.$$

The length through which the rays were exposed to the magnetic force was 8 c.m. If in this distance they were bent through the above angle, the radius of curvature would be—

$$r = \frac{8}{0{\cdot}000405} \text{ c.m.} = 19{,}800 \text{ c.m.}$$

The strength of the magnetic field was determined in the usual manner, by observing the throw of a galvanometer when a small coil of known dimensions connected up with it was suddenly withdrawn from the region between the pole pieces. To reduce the results to absolute measure, the throw due to reversal of an earth-inductor in the same circuit was observed.

In this way the strength of the field was found to be

3270 C.G.S.

It is convenient to exhibit the result by giving the maximum field which the experiments indicate as unable to produce a curvature of radius 1 c.m.

Since a field of 3270 does not produce a curvature of radius less than 19,800 c.m., we see that the field required to produce a curvature of radius 1 c.m. cannot be less than

$$6{\cdot}5 \times 10^7.$$

Owing to the fact that the magnetic field was *reversed* instead of being merely shut off, the experiment is really of double the sensitiveness indicated above. But, in order to be well on the safe side, it has been thought best to leave this out of account.

For the sake of comparison I have attempted a rough estimate of the amount of the magnetic deflection of the Becquerel rays. The method employed was as shown in Fig. 1.

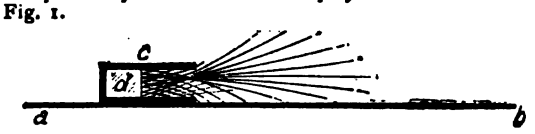


Fig. 1.

A photographic plate, shown in section at *a b*, was lai on the top of the square pole-pieces of a magnet, the magnetic force being perpendicular to the plane of th diagram. The plate was covered with thin aluminium foil; *c* is a metal capsule filled with the substance *d*, which emitted the rays.*

When no magnetic force was acting, the rays were emitted from the capsule as indicated in Fig. 1, some of some of them striking obliquely on the plate. On develop ment after one hour's exposure, a shadow was obtaine beginning at the edge of the capsule *c*, and extending a short distance. The effect gradually tailed off, and at a few c.m. distance away from *c* it was inappreciab le When the magnetic force was in such a direction as to bend the rays down into the plate (Fig. 2), the shadow extended further. When, on the other hand, the magnet

* The substance employed was a preparation from uranium residues, supplied by de Haen, Hamburg.

was reversed so as to bend the rays away from the plate (Fig. 3), the shadow obtained on development was much

FIG 2.

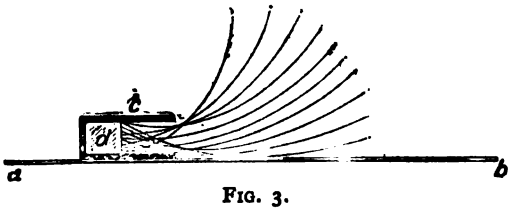


FIG. 3.

shorter, the time of exposure being, of course, in each case the same.

The numerical estimate of the curvature of the rays was obtained from an experiment of the latter kind.

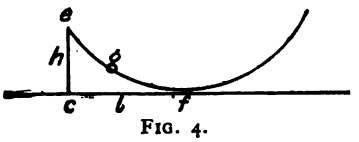


FIG. 4.

Let us suppose that ec (Fig. 4) represents in section the front surface of the radiating substance, cf the surface of the photographic plate.

Let f be the place furthest from c at which the darkening on the photographic plate was perceptible. Now the rays which reach furthest are those which proceed from e, the highest point of the radiating surface, as may easily be seen from geometrical considerations. The rays which reach f must consequently proceed from e. Rays proceeding from any lower point of the surface will either be bent up so as never to reach the plate at all, or else they will strike it short of f. The ray which reaches f from e will clearly just graze the surface of the plate at f.

If r be the radius of curvature, b the distance ec, and l the distance cf, then—

$$h(2r - b) = l^2, \text{ or } r = \tfrac{1}{2}\left(\frac{l^2}{h} + h\right).$$

If then we measure b, the height of the highest part of the radiating surface above the plate, and l the greatest distance to which the darkening of the plate extends, we have data for determining r.

It must be admitted that the measurement of l involves great uncertainty. The image gradually tails off, and any estimate of its length must to a great extent be arbitrary.

The value of r deduced is more uncertain still, since l^2 is involved in calculating it. But, in spite of these objections, the method may, I think, be relied on to give the order of magnitude of r, and that is all that is required, so far as the conclusions which it is here sought to draw are concerned.

In one experiment the length l was estimated at 2 c.m.; b was 0·8 c.m. Thus $r = 3$ c.m. approximately.

The strength of the magnetic field, measured as before, was 1680 C.G.S. Thus the field required to produce a curvature of radius 1 c.m. is about 5×10^3.

In another experiment, l was 1·8 c.m., b was 0·8 c.m., and the field 2140. This gives practically the same result as the preceding.

In an experiment described by Professor J. J. Thomson, a beam of cathode rays was bent to a radius of curvature of 9 c.m. in a field of 35 units. Thus a field of 315 would have been required to bend it to a radius of 1 c.m.

Let us now collect the results obtained, and compare them with this.

The field which would be required to produce a curvature of 1 c.m. radius would be—

For cathode rays	3×10^2
„ Becquerel rays	5×10^3
„ Röntgen rays not less than ..	6×10^7.

If the Röntgen rays are magnetically deflected at all, it is by an amount less than a ten-thousandth part of that observed in the case of the Becquerel rays.

The magnetic deflectibility of the Becquerel rays cannot but be considered to be a most characteristic property. And the above result appears to make it tolerably certain that the Röntgen rays do not possess this property. It is to be concluded, therefore, that the Becquerel rays are, after all, essentially different in character from the Röntgen rays.

ON GADOLINIUM.*

By C. BENEDICKS.

(Continued from p. 53)

Identity of Gadolinium.

DOUBTS have been cast on the elementary character of gadolinium. Delafontaine imagined Marignac's Yα to be a mixture of the terbium and decipium earths. This opinion was shown by Cleve, in 1885, to be untenable, since pure gadolinium earth is nearly colourless, whilst that of terbium earth is orange-coloured. As regards decipium earth, its identity with that of gadolinium might eventually be established, for the only differences pointed out by Delafontaine are the atomic weight 171 and the insolubility of the potassium double sulphate in saturated potassium sulphate solution. The reliability of the information concerning decipium earth is still questionable.

Sir W. Crookes, who himself prepared gadolinium earth from samarskite and made a searching study of its phosphorescent spectrum, concludes that three components are present in gadolinium. In consideration of the great delicacy of his method, and the less completely known relations which influence it, this should be recognised as a most valuable aid to the chemistry of the future, though, on the other hand, it scarcely promises great possibility of separation.

Demarçay's statement about an element coming next to samarium might, to a certain extent, be considered as a splitting up of gadolinium. For this hypothetical element, whose oxides and salts are to be colourless as should be those of gadolinium, would only be distinguishable from gadolinium by the greater solubility of the nitrate in concentrated nitric acid. The only foundation for belief in its existence is the spark spectrum. Demarçay has announced no atomic weight determinations of the different nitrate fractionations. In consequence, I have carried out determinations with material treated according to Demarçay's statements. These determinations only indicate that the atomic weight in the first fractions was slightly lower than in the last fraction II., with which the atomic weight determinations were made, the reason probably being that a small amount of samarium was still present in the first fractions.

By observation of the spark spectrum of fraction II., I could discover no E line. Still more conclusive evidence against the presence E_2O_3 in my material lies in the fact that it was re-crystallised ten times from concentrated nitric acid, by which means, according to Demarçay, E_2O_3 must have been removed. If it appears later that E_2O_3 is a fundamental substance of remaining elements, it is probable that it is absent in my material.

* *Zeitschrift für Anorganische Chemie*, xxii., 5, p. 393.

Spectrum.

The gadolinium spectrum has been studied and described by Lecoq de Boisbaudran. With reference to this, it appears that two eminent experimentalists, Thalen and Bettendorff, were unable to get a spectrum. The cause is to be found in the differences in the methods of procedure. Thalen used aluminium poles, covered with a chloride solution. By using a Salet tube in conjunction with an induction coil with a long secondary wire, one can obtain without difficulty a beautiful spectrum, which consists of bands and bright lines.

This spectrum, according to Lecoq, is specially beautiful when a Demarçay induction coil with a short secondary coil is used, the poles being placed as far apart as possible; with a short distance between the poles quite a different spectrum is obtained, consisting only of bright distinct lines.

Demarçay also announces a few Gd lines in the violet part of the spectrum.

There is no absorption band in the visible part of the gadolinium spectrum, but, according to Soret, there is an absorption band from λ 280 to λ 245 in the ultra violet.

Compounds of Gadolinium.

Gadolinium Oxide, Gd_2O_3, the only oxide of this element, forms a white powder; a scarcely visible tinge in the yellow is probably caused by a minute trace of terbium oxide. It is easily soluble in acids; the reaction is slight at first, but on standing or through warming becomes very powerful. The oxide absorbs CO_2 quickly from the air, and is rather hygroscopic. The oxide is not reduced by heating in hydrogen, but, as Bettendorff has already discovered, a small quantity of a darker coloured earth is produced, which is somewhat less soluble in acids, but which has exactly the same atomic weight as the remaining earth, as determined by Bettendorff.

Specific gravity	I. 7·405 at 16°
,, ...	II. 7·407 at 15°
Molecular volume	48·6.

Gadolinium hydroxide is a gelatinous precipitate, rapidly absorbing CO_2 from the air.

The salts of gadolinium are, with few exceptions, colourless; they have no absorption spectrum, and possess a sweet astringent taste. They resemble the salts of yttrium in these and in most other properties, as well as in their behaviour with regard to different reagents.

Gadolinium Chloride, $GdCl_3+6H_2O$.—Quadratic double pyramids, optically uniaxal, crystallising well in thick blunt plates. Crystallises from neutral solution over sulphuric acid in rather large, somewhat deliquescent crystals, and very easily from hydrochloric acid solution in smaller crystals, which contain the same quantity of water of crystallisation.

Analysis of $GdCl_3.6H_2O$.

	I.	II.	III.	IV.	
Gd ..	42·2	41·94	42·00	41·70	per cent.
Cl_3 ..	—	—	28·46	28·49	,,
Calculated percentages			Gd	42·10	per cent.
			Cl	28·75	,,

Mean specific gravity .. .. .. 2·424
Molecular volume .. .. .. 152·8

Gadolinium Bromide, $GdBr_3+6H_2O$.—Crystallises from the solution of the oxide in hydrobromic acid over sulphuric acid in small, pointed, rhombic plates, coloured brown by the presence of free bromine.

Analysis of Salt.

Gd ..	30·70 per cent.	Calculated	30·95 per cent.
Br_3 ..	—	,,	47·62 ,,

Mean specific gravity .. .. .. 2·844
Molecular volume .. .. .. 177·2

Gadolinium Platinichloride, $GdCl_3+PtCl_4+10H_2O$.—A quantity of this, together with some $GdCl_3$, crystallises from equivalent quantities of $GdCl_3$ and $PtCl_4$. Orange-yellow, prismatic needles. Fairly stable in air, but affording no measurement.

Analysis of Salt.

Gd ..	19·90 per cent.	Calculated	20·03 per cent.
Pt ..	25·0 ,,	,,	25·01 ,,
Cl_4 ..	—	,,	31·86 ,,

Specific gravity .. .. 2·719
Molecular volume .. .. .. 286·5

Gadolinium Aurochloride, $GdCl_3+AuCl_3+10H_2O$.—Crystallises from the mixed solution of both salts ($AuCl_3$ in excess) in beautiful yellow plates after much concentration. The salt loses H_2O over sulphuric acid.

Analysis of Salt.

Gd ..	21·00 per cent.	Calculated	20·91 per cent.
Au ..	26·00 ,,	,,	26·44 ,,
Cl_3 ..	—	,,	28·52 ,,

Specific gravity .. .. 2·706
Molecular volume .. .. .. 275·7

Gadolinium Platinocyanide,—

$$2Gd(CN)_3+3Pt(CN)_2+18H_2O,$$

was obtained by double decomposition of calculated quantities of gadolinium sulphate and barium platinocyanide and concentration of the filtered solution over sulphuric acid. Beautiful cherry-red rhombic crystals, some long and pointed, some tabular and flattened, which possess a bright metallic lustre on the prismatic and pyramidal surfaces. The salt is isomorphic with the corresponding yttrium and erbium compounds. As the angular differences between gadolinium on the one hand, and yttrium on the other, are very trifling, and the variations of individual measurements are often greater, I have not calculated the constant, as later in the case of other salts, but give only the corresponding angular measurements of the yttrium compounds, as they appear from the researches of Dr. H. Topsoë:—

	No. of crystals.	Edges.	Observed.	(Topsoë).
$\bar{1}10 : 110$	7	8	83° 27′	83° 28′
$\bar{1}10 : 1\bar{1}0$	6	8	96° 27′	96° 32′
$001 : 1\bar{1}0$	17	20	88° 24′—91° 40′	90° 0′
$001 : 221$	2	2	61° 44′	61° 36′
$\bar{1}10 : 221$	7	4	28° 36′	23° 24′

The optical orientation is the same as in the yttrium compound.

The crystals are not so stable in air as those of the yttrium and erbium compounds, but gradually decompose and become yellow.

Analysis of $2Gd(CN)_3+3Pt(CN)_2+18H_2O$.

Gd_2 ..	20·53 per cent.	Calculated	20·36 per cent.
Pt_3 ..	38·08 ,,	,,	38·14 ,,
CN_2..	—	,,	20·36 ,,
$18H_2O$	—	,,	21·24 ,,

Specific gravity .. .. 2·563
Molecular volume .. .. .. 597·9

As already stated, this salt is isomorphous with the corresponding yttrium and erbium salts; the fine red colour and the green metallic lustre are common to all these compounds. The platino-cyanides of the cerium metals (to which samarium is allied) are, on the contrary, yellow with a blue metallic lustre, and crystallise in monoclinic prisms. Gadolinium therefore belongs to the yttrium group. But the water of crystallisation of the gadolinium compound is $18H_2O$, like that of cerium, whereas that of the yttrium and erbium salts is $21H_2O$.

(To be continued).

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

Part II.

TUNGSTEN AND MOLYBDENUM.

By HARRY BREARLEY.

(Continued from p. 50).

TUNGSTEN (*continued*).

II. Gravimetric Estimations.

241. Bernoulli (*C. N.*, v., 116).—Fuse insoluble tungstates with soda carbonate; neutralise extract with acetic acid, precipitate with lead acetate, digest $PbWO_4$ in ammonium sulphide, evaporate filtrate with HNO_3, and weigh WO_3. By treating the nearly dried nitric solution with ammonia, niobic acid is left undissolved.

242. Zettnor (*C. N.*, xvi., 12).—By precipitating with lead acetate from a faintly acetic acid solution (see 243).

243. Brearley (*C. N.*, lxxix., 66). — The precipitate formed in acetic solutions with lead acetate is an indefinite acid tungstate of lead containing all the W. On dissolving in strong HCl and diluting, all the WO_3 is re-precipitated. $PbWO_4$ is soluble in alkaline acetates and chlorides, but not in nitrates. True $PbWO_4$ is formed in faintly ammoniacal solution, in presence of $(NH_4)NO_3$, by a few minutes' boiling.

243*a*. Kern (*C. N.*, xxxv., 67).—WO_3 and SiO_2 separated from acid solution of iron, washed with HCl and alcohol. Separate SiO_2 with ammonia, and precipitate WO_3 from caustic filtrate with HCl.

244. Parry and Morgan (*C. N.*, lxvii., 260).—Acid solution of the steel is evaporated, but not baked. Filtered SiO_2 and WO_3 separated with ammonia (see 219).

245. Kemery (*J. I. S. I.*, 1894, i., 614).—Dissolve in nitro-sulphuric acid, evaporate to SO_3 fumes, separate SiO_2 with HF.

246. Schneider and Lipp (*C. N.*, li., 297).—Steel attacked with Br and HNO_3; evaporated; residue not soluble in dilute HNO_3 fused and evaporated with HNO_3; SiO_2 and WO_3 separated with $KHSO_4$ and $(NH_4)_2CO_3$.

247. Vienna Assay Office (*J. I. S. I.*, 1884, 230).—Decompose with aqua regia or bromine; separates SiO_2 as in 246.

248. Perillou (*J. I. S. I.*, 1884, 629).— Dissolve in HNO_3, evaporate, re-dissolve, boil two hours, weigh WO_3+SiO_2, and heat to redness in dried HCl gas. The tungsten is volatilised (see 288); residual silica weighed.

249. Schneider (*J. I. S. I.*, 1885, 245).—Iron alloys treated with HCl repeatedly. Only traces of tungsten pass into solution from alloys rich in tungsten.

250. Namias (*J. C. S.*, lxii., 539).—Rich alloys digested with Br in an alkaline solution and then evaporated with HCl. WO_3+SiO_2 ignited in hydrogen (see 223); the loss in weight being calculated to WO_3. Steel digested with HCl in absence of air; what little passes into solution is recovered by evaporating with sugar; charred residue contains WO_3+SiO_2.

251. Preusser (*C. N.*, lx., 37).—In rich alloys, powdered sample ignited to WO_3, evaporated with acids, residue fused with soda carbonate, extract precipitated with HCl, SiO_2 separated with ammonia, $(NH_4)_2WO_4$ evaporated and ignited to WO_3. Notes the solubility of SiO_2 in ammonia.

252. Ziegler (*C. N.*, lx., 272).—Points out that tungsten attacks platinum, probably forming an alloy. Throws the W powder on to fused $(NH_4)NO_3$, which effects a superficial oxidation, and is volatilised.

253. Wdowiszewski (*J. I. S. I.*, 1895, ii., 597).—The FeW alloy fused with borax glass and NaK carbonates; water extract evaporated with HCl; washing with dilute $(NH_4)NO_3$ prevents WO_3 passing through the filter.

254. Cremer (*J. S. C. I.*, 1895, 679).—Fuse the ore with pyrosulphate ($K_2S_2O_7$), dissolve in dilute HCl, add cinchonin, and wash WO_3 with cinchonin solution. Separate SiO_2 with ammonia.

255. Ziegler (*J. I. S. I.*, 1890, i., 373).—Fuse with soda nitrate, evaporate extract with acid, separate SiO_2 and WO_3 with HF. Any W in the fusion residue is separated and precipitated with $HgNO_3$.

256. Ziegler (*J. S. C. I.*, 1890, 216).— Behaviour of reagents with metallic W. Oxidation of a sample gauged by dusting into Na_2CO_3 kept fluid so that the W may sink promptly. Steel is attacked with nitro-sulphuric acid. A detailed account of the process 255 (see also *J. S. C. I.*, 1891, 388).

257. Helmhacker (*J. S. C. I.*, 1896, 656).—Wolfram ore fused with soda carbonate; WO_3 precipitated from filtrate with acids; and SiO_2 and SnO_2 separated with HF and KCN fusions respectively.

258. Carnot and Goutal (*J. C. S.*, lxxii., 521).—Iron separated with copper solution. Residue freed from SiO_2, fused, and precipitated with $HgNO_3$.

259. Schöffel (*C. N.*, xli., 31; and *J. I. S. I.*, 1880, 345). — Separate iron with solution of copper-ammonium chloride, and precipitate fused residue with $HgNO_3$. SiO_2 separated by fusion with $KHSO_4$. Roast alloys of over 12 per cent W, and fuse. Dissolve steels in aqua regia, dilute, and allow to stand one to two days.

260. Von der Pfordten (*C. N.*, l., 18).— Precipitation with $HgNO_3$ (Berzelius's method) or evaporation with HCl.

261. Smith and Bradbury (*J. C. S.*, lxii., 241).—Tungsten may be estimated by means of its Ba, Pb, or Cd salts, but not effectually by means of the Sr, Ca, Ag, Co, or Bi salts.

262. Lefort (*C. N.*, xlv., 57).—To slightly acetic solution of a soluble tungstate add quinine acetate or sulphate. Destroy quinine by igniting and treating with HNO_3. Method exact.

263. McKenna (*J. S. C. I.*, 1898, 1185).—FeW alloys decomposed with soda peroxide in Cu crucible; WO_3 precipitated by evaporation with HCl; Cr and Al are separated by $(NH_4)HO$. Carbon is determined by fusing with peroxide and estimating the carbonates formed.

264. Philipp (*J. S. C. I.*, 1882, 152).—Powdered tungsten bronze heated with an ammoniacal solution precipitates Ag equivalent to the oxygen needed to completely oxidise the compound. The NaO and WO_3 are determined in the filtrate.

264*a*. Ibbotson and Brearley (*C. N.*, lxxx., 294).—Indirect process for estimating W in metallic powders depending on the solubility of the oxidised metal in NaHO. Results exact.

III. Volumetric Estimations.

265. Hundeshagen (*J. C. S.*, lxviii., 85).—WO_3 separated as usual, is dissolved in NaHO, and excess titrated.

266. Hitchcock (*J. C. S.*, lxx., 526).—Uranyl nitrate or chloride precipitates W completely. A volumetric estimation, using ferrocyanide as indicator, is based on this fact. (See also 294).

IV. Detection and Miscellaneous Notes.

267. Mallet (*C. N.*, xxxi., 276).—A dilute HCl solution of an alkaline tungstate treated with sulphocyanate and some zinc assumes a fine amethyst colour. (See 300).

268. Defacqz (*C. N.*, lxxiv., 88).—Zn or Al in an acidified solution of a tungstate gives blue colour. The material is got into solution with bisulphate and H_2SO_4: triturate with phenol (red colour) or hydroquinone (violet). Other organic reactions given.

269. Langley (*J. I. S. I.*, 1893, i., 373).—By the use of an emery wheel, less than ½ per cent W can be detected in steel; it gives only a dull fire instead of the brilliant showers of sparks.

270. Skey (*C. N.*, xiv., 256).—WO_3 suddenly refrigerated assumes a black colour permanent in air. Other new reactions.

271. Skey (*C. N.*, xvii., 157).—Recently ignited charcoal shaken up with a sulphuric solution of tungsten absorbs the W, but gives it up to caustic alkali.

272. Carnot and Goutal (*J. C. S.*, lxxii., 555).—Fe_3W and Fe_3Mo_2 are left undissolved by dilute acids acting on iron; these bodies therefore exist in steel.

273. Moissan (*J. C. S.*, lx., 607).—Tungsten is readily dissolved by a mixture of HF and HNO_3.

273*a*. Burghardt (*C. N.*, lxi., 260).—Wolframite is decomposed by mixing with charcoal and projecting into fused soda. WO_3 precipitated from filtrate by making slightly acid with HCl. Chrome iron ore is similarly decomposed.

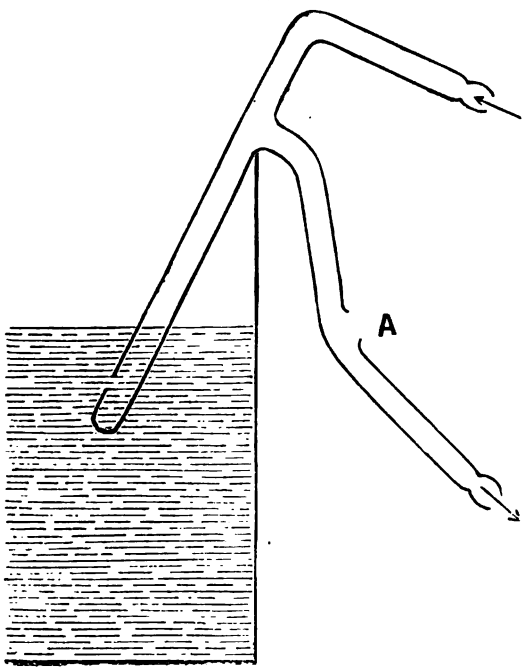
274. Riddle (*J. I. S. I.*, 1890, i., 365).—Powdered metallic tungsten in contact with concentrated HCl for three days was not at all dissolved.

(To be continued).

NEW FORM OF WATER-BATH REGULATOR.

By H. STAFFORD HATFIELD.

A simple device applicable to any vessel is often required for maintaining for some time without attention a constant level of liquid in a water-bath or similar apparatus. Some form of syphon is usually employed, connected with



a reservoir, maintained at constant level by inflow and outflow of water. But all such regulators cease to work in a short time if ordinary tap-water is used, because the heating of the inflowing water during its passage down the syphon-tube fills the latter with air, and so the supply ceases, unless an air-collector of considerable capacity is sealed on at the highest point of the syphon.

The annexed figure illustrates a form of apparatus which renders unnecessary a reservoir, and which is not stopped in its action by air-bubbles. The supply-tube bifurcates, and one limb dips under the liquid in the bath. The other is opened to the air at A. The water in the bath will keep at the level of A, or slightly above it; the excess of level being due to the force necessary to divert the stream of water. All the tubes must be full of water, but air-bubbles on rising are immediately swept away by the current of water, which must be sufficient to do this properly, but need not be great. The water must leave the sides of the glass at A. The tubes may be bent to fit any particular form of vessel. After a thorough test, I am satisfied of its efficiency.

University College, London.
January 25, 1900.

THE ANALYSIS OF GLUE.

By R. WOY.

The methods now in vogue for the analysis of glue are those of R. Kissling (*Moniteur Scientifique*, March, 1899, p. 222).

According to Kissling, the proportion of fatty matters should vary from 0·001 to 0·09 per cent. Now, by making use of a method described by Hilger, very analogous to the so-called "gypsum" method for the extraction of alkaloids, the author found 0·24 to 0·67 per cent.

The sheets of glue are broken up in a covered mortar, into sufficiently small pieces, and 20 grms. are weighed out and placed in a porcelain crucible; to this are added 40—50 c.c. of boiling water, and 5—10 c.c. of hydrochloric acid to decompose the traces of soap which may be present in the substance. The glue is dissolved by gently heating with continuous stirring. A suitable quantity of gypsum is then added, a little at a time, until the mass becomes first pasty and then solid. The sides of the crucible are then scraped, and the whole dried for two to three hours at 105—110°.

The powder thus dried is placed in a small flask and exhausted with ether for five or six hours in a Soxhlet extractor. Finally, the ether extract is dried in an oven.

For the complete analysis of glue we dissolve 50 grms. of the sample by repeated treatments with water (200 c.c. at a time); the solution is made up to 1 litre and well mixed.

Estimation of the Water.—Evaporate 25 c.c. of the solution in a crucible, as in the estimation of the dry extract of wines, and dry the residue at 110° for three hours.

Ash.—The residue furnished by the estimation of the water is carbonised, the residue is moistened with alcohol and ground up, the alcohol is carefully driven off by heat and finally incinerated.

Nitrogen.—25 c.c. of the solution of glue are treated by Kjeldahl's method. To effect its decomposition the author used 20 c.c. of a mixture of three volumes of concentrated sulphuric acid, and two volumes of fuming sulphuric acid. To these 20 c..c of acid there are added 10 grms. of anhydrous sulphate of potash and 2 grms. of anhydrous sulphate of copper.

Pure glue contains about 18·5 per cent of nitrogen.

Acidity.—100 or 200 c.c. of the solution of glue are diluted with water, and titrated with decinormal soda, using phenolphthalein as an indicator. We then add an excess of decinormal hydrochloric acid, and titrate again with soda. The acidity is expressed in c.c. of normal acid per 100 c.c. of glue.

Free Volatile Acids.—These are determined by distilling 20 to 30 grms. of glue in a current of steam, and titrating the distillate with normal soda.

Combined Volatile Acids.—These are estimated in the same manner, but sulphuric acid is added before distillation.

Sulphurous Acid.—Distil in a current of carbonic acid, collect the product of distillation in a solution of iodine, and precipitate with chloride of barium.

Finally, the author gives it as his opinion that, in the estimation of glues, chemical analysis is much to be preferred to mechanical tests, the results of which are always doubtful and liable to contradiction.—*Zeits. f. Offentl. Chem.*, iv., p. 755.

ON THE HEAT OF OXIDATION OF TUNGSTEN.

By M. DELÉPINE and L. A. HALLOPEAU.

We decided to measure the heats of combustion of tungsten and of its lower oxide, WO_2, for the purpose of comparing them with those of the other metals, and of assigning, in this respect, a place to tungsten in the series of the elements; the results obtained have led us to make certain experiments necessary to justify the position assigned to it by us.

Tungsten, W. — We only succeeded in obtaining satisfactory results after several failures. Combustion in oxygen at the ordinary pressure, combination with a halogen element, attack by water or by an acid, displacement by another element of which the heat of oxidation is known, &c., generally used for the determination of the heat of formation of an oxide, are not applicable to tungsten, or to its oxide, WO_2, since tungsten does not burn in oxygen or chlorine unless it is heated continuously, and no other reactions into which it enters are sufficiently rapid; all these conditions are incompatible with the use of the calorimeter. It then occurred to us to make use of M. Berthelot's calorimetric bomb.

The finely divided tungsten (0·8 to 1·0 grm.) must be ignited by means of a little camphor (0·01 to 0·03 grm.), the camphor being set alight by a few grains of gun-cotton (0·01 grm. at the most), which is set fire to by means of a platinum wire brought to incandescence by means of an electric current. Thanks to this series of successive ignitions the spiral of iron wire, which might bring into the calculation the formation of an unknown quantity of tungstate of iron, is done away with.

It is also necessary to avoid the use of platinum; in our first experiment the tungsten burning in compressed oxygen (25 atmos.) pierced the crucible over a surface of several square c.ms., the temperature being so high; possibly an alloy is formed.

Happily we placed a layer of water covered with a sheet of platinum at the bottom of the bomb; this sheet of platinum was also pierced, but the bomb was not injured. We were careful always to take the same precautions in our future experiments. Porcelain also cannot be used; it is always partially melted, and is even sometimes pierced. We finally decided to use the bottoms of clay crucibles of a uniform thickness, 6 to 7 m.m. in height and 10 to 15 m.m. in diameter.

Even then success did not always follow, but we were able to obtain sufficiently concordant figures in three experiments made with pure tungsten; that is, per grm., 1049, 1044, and 1093 cal., a mean of 1062 cal.

The first of these figures carries a slight correction, established by the determination in the product of combustion, the quantity of fresh oxygen capable of combination (0·0027 grm.); this brought the original figure from 1036 to 1049 cal.; in the two other experiments we were not able to fix any fresh oxygen.*

* If instead of burning pure tungsten we burn carburised tungsten, we obtain much higher figures; thus, a melted sample that we burnt gave off 1287 cal. per grm., which points to a proportion of 3 per cent of carbon present. In fact, on analysis this tungsten did not give the correct amount of tungstic anhydride.

Binoxide, WO_2.—Our binoxide was a magnificently crystallised body. Its combustion offered none of the preceding difficulties; we were able to use a platinum crucible, and instead of operating at 25 atmospheres on 1 grm., we operated at 15 atmospheres, and even less, on 3 grms. of binoxide at a time. The combustion of each grm. of binoxide gave off: 292·8, 304·2, 293·1, 306·6 cals., or an average of 299·2 cal.

It results from these figures and from the preceding ones that—

$W + O_3 = WO_3 + 195{\cdot}41$ cal. at constant volume $+196{\cdot}3$ cal. at constant pressure.

$WO_2 + O = WO_3 + 64{\cdot}63$ cal. at constant volume $+64{\cdot}9$ cal. at constant pressure.

$W + O_2 = WO_2$ cal. at constant volume $= 131{\cdot}4 = 2 \times 65{\cdot}7$ cal. at constant pressure.

The fixation of each atom of oxygen thus gives off nearly the same quantity of heat when combining with the metal or the oxide already formed; but if we are correct in regarding the heat of formation of WO_2 as exact to 1 cal., we are led to believe in too small a heat of oxidation for W into WO_3.

Nevertheless, these results enable us to make a preliminary comparison with the heat of oxidation of a few bodies, with regard to the fixation of 1 atom of oxygen. The following table, which carries with it all the consequences mentioned later on, gives the amount of these heats of oxidation:—

	Cal.
Mg	143·4
Al	131·0
Alkaline and alkaline-earthy metals	> 90·0
Si	89·8
Zn	84·8
Sn	70·7
$H(H_2O$ liq$)$	69·0
W	65·4
Fe into FeO	65·7
Fe into Fe_2O_3	65·2 × 3
Fe into Fe_3O_4	67·7 × 4
$SK_2 + O_4$	60·2 × 4
$H(H_2O$ gas$)$	58·3
Sb_2 into Sb_2O_3	55·6 × 3
Pb	50·8
Cu_2 into Cu_2O	43·8
Hg, Pb, Ag, Au	< 25·0

We know that aqueous vapour oxidises tungsten at a relatively low temperature, but that, inversely, hydrogen completely reduces tungstic anhydride at a high temperature, a result comparable with the action of H_2O and H on iron and its oxides, which tungsten resembles by its heat of oxidation and by the equality of the successive disengagements of heat in its different degrees of oxidation.*

We also know that tungsten does not decompose either silica, magnesia, or alumina; neither does it decompose the anhydrous oxides of the alkaline or alkaline-earthy metals, as may be seen in the electrolytic preparation of the tungstates, when at a certain time they may be left in prolonged contact after the bath has become alkaline. On the other hand, magnesium gave with WO_3 a reaction of extreme violence. We did not think it worth while to try the action of the alkaline metals, but zinc gave a very important and perfectly regular reaction by which we were enabled to prepare tungsten.

The action of tin on the oxides themselves was not studied by us; but we found that, conformably with its great heat of oxidation, it brings the alkaline tungstates

* Water, as liquid, does not oxidise tungsten, but W and WO_2 decompose it with facility in the presence of alkalis without there being any reverse action following the formation of a dissolved tungstate—

$W + 2KOH$ dis. $+ 2H_2O$ liq. $= WO_2K_2$ dis. $+ 3H_2$ . . $+(x - 1{\cdot}9)$ cal.

$WO_2 + 2KOH$ dis. $= WO_2K_2$ dis. $+ H_2$ . . . . . $+(x + 4{\cdot}7)$ cal.

[x = the heat of hydration of $4WO_3$ changed into metatungstic acid, $(WO_3)_4H_2O$].

to the state of bronzes, and even separates metallic tungsten.

Inversely we found that tungsten easily decomposes the oxides of antimony, lead, and copper, and sets the metal of these oxides free, at the temperature of a simple gas-burner. It was therefore useless to examine the reduction of oxides, of which the formation gives off still less heat.

Tungsten changes the sulphates into sulphides, a result which conforms with the figures in the table.

Finally, its tendency to pass to the state of tungstic oxide may be made use of on the solutions of the last oxides in the table; the blue (W_2O_5) or red (WO_2) solutions contain the lower oxides of W, and reduce to the lower state of oxidation the cupric, mercuric, and platinic salts, and precipitate the metal in auric and argentic solutions; at the same time the blue or red colour disappears on account of the transformation to WO_3.

The place assigned to tungsten according to the heat of oxidation, 3×65 cal. (about), seems to us to be amply justified by the preceding experiments.—*Bull. Soc. Chim.*, Series 3, xxi., No. 22.

ON THE COLOUR OF CARBIDE OF CALCIUM.

By HENRY MOISSAN.

We have shown in previous papers, that the carbides of potassium, C_2K_2, and of lithium, C_2Li_2, can be prepared in the form of transparent colourless crystalline plates. We have also obtained carbide of aluminium, C_3Al_4, in the form of well-crystallised transparent yellow plates. On the other hand, when we first prepared the carbides of calcium, strontium, and barium in the electric furnace, we described these new crystalline compounds as only being slightly transparent in very thin plates, and having a characteristic reddish brown appearance.

This reddish brown appearance and the colouration of the crystals are only due to an impurity. When carbide of calcium does not contain a trace of iron, it is quite transparent, like carbide of lithium or chloride of sodium.

We will give as examples the following experiments:—

1. When we heat metallic calcium to a dull red heat* in a crucible brasqued with pure amorphous carbon, obtained by the rapid decomposition of acetylene, we obtain a white carbide of calcium, decomposable by cold water, forming slaked lime, and giving off acetylene. The carbide, examined under the microscope, is found to consist of a mass of transparent crystals.

2. When we heat pure hydride of calcium, CaH_2, of which we have described the preparation by the direct union of hydrogen and calcium (1 : 1) in a brasque of pure amorphous carbon, we obtain a white carbide of calcium, C_2Ca, in thin plates, which are quite transparent.

3. The result is identical when we repeat the previous experiment with crystallised nitride of calcium, N_2Ca_3.

4. Acetylenic ammoniacal acetylide of calcium, $C_2Ca.C_2H_2.4NH_4$, which we obtained at 60° by the action of acetylene on calcium-ammonium, leaves, on its complete decomposition, a carbide of calcium, C_2Ca, of extreme whiteness, and which is quite transparent under the microscope.

Finally, we will give the following experiment:—We took this white carbide of calcium and mixed it with a quantity of oxide of iron; the whole was then melted in the electric furnace in a crucible of pure graphite. After cooling, we obtained a reddish brown carbide of calcium, exactly like the carbide prepared commercially.

Absolutely pure carbide of calcium is therefore transparent; when it has a reddish brown appearance, this should be attributed to the presence of iron. A trace only of this metal is sufficient to produce the colouration.—*Bull. Soc. Chim.*, Series 3, vol. xxi., No. 22.

* This synthesis of calcium carbide takes place at a low temperature; it can be done by heating a mixture of calcium and carbon in a porcelain crucible over a spirit flame. We must add, however, that the heat given off by the union of the carbon and the calcium is sufficiently great to bring about the fusion of the carbide of calcium—a fusion which has, up to the present, only been attained by means of the electric furnace.

ANTIMONIC ACIDS AND ANTIMONIATES.

By A. E. DELACROIX.

In a preceding paper (*Journ. de Pharm. et de Chim.*, (6), vi., pp. 337 and 533), I proved the existence of two soluble antimonic acids. As they have nothing in common with pyro- and ortho-phosphoric acids, I propose calling them tetra-antimonic and tri-antimonic acids, names which are in accordance with the facts.

The first gives acid or monobasic salts $(Sb_2O_5)_4M_2O$, and neutral or bibasic salts $(Sb_2O_5)_3M_2O$.

The second gives monobasic salts $(Sb_2O_5)_3M_2O$, neutral or bibasic salts $(Sb_2O_5)_3(M_2O)_2$, tribasic salts $(Sb_2O_5)_3(M_2O)_3$, and other complex salts.

Tetra-antimonic Acid.—The crystallised hydrate of this body is prepared by completely freezing a solution of the acid. The ice is melted in alcohol, and thin flakes are collected, acting on polarised light. After desiccation in air, which takes place fairly rapidly, this hydrate is found to be $Sb_2O_5,4H_2O$. Found—water, 18·81; calculated—water, 18·36. Over sulphuric acid it becomes $Sb_2O_5,3H_2O$, and at 100°, $Sb_2O_5,2H_2O$. It answers to the amorphous hydrate of Daubrawa (*Bull. Soc. Chim.*, xxix., 118).

Tetra-antimoniates.—The alkaline salts are soluble in water, but insoluble in water containing other salts. The bibasic salts are produced by the neutralisation of the acid by the alkaline bases. Alcohol precipitates them from this solution; this does not take place with the monobasic salts. After precipitation with alcohol, the precipitate is separated by rapid filtration, and set to dry on a porous plate. The acetates may be treated in the same manner, but a slight excess must be used. Thus prepared, these salts are amorphous. Crystallised salts, having the appearance of the hydrate described above, are obtained by freezing antimonic solutions to which suitable quantities of bases or salts of alkaline metals have been added. Desiccation makes them insoluble. The alkaline and alkaline-earthy antimoniates turn yellow on calcination, which appears to decompose them. The other tetra-antimoniates are prepared from the acetates, of which it is better to use dilute solutions, to prevent the formation of compact masses which are difficult to wash. They are insoluble in water, soluble in concentrated HCl, more or less soluble in tetra antimonic acid.

Certain tetra-antimoniates split up, in the presence of water, into mono and bibasic triantimoniates.

As the hydrate and tetra-antimonic acid both change easily into triantimonic compounds, it is necessary to make certain of the condition of the solutions used to make the tetra-antimoniate. To effect this, the equivalent is taken in 1/10 normal soda of—(1) the last mentioned tetra-antimonic solution containing HCl; (2) of the same solution transformed into triantimonic acid; and (3) the weight per litre of Sb_2O_5 is determined; this may be done with sufficient accuracy by evaporating down a known volume, and weighing the calcined residue. It is easy to see that the difference of the two titrations $\times 3$ = the value of the tetra-antimonic acid of Sb_2O_5, in solution in N/10 soda, on condition, of course, that this Sb_2O_5 was entirely in the tetra- state.

These data suffice for us to calculate in what proportion, the two antimonic acids existed in the original solution, and also how much hydrochloric acid was also present.

Bibasic Tetra-antimoniate of Soda $(Sb_2O_5)_2NaO_2,9H_2O$.—This salt crystallises in flakes, and is prepared by freezing the acid exactly neutralised by soda. Found—Na_2O, 7·39; Sb_2O_5, 73·42; H_2O and loss, 19·18.

Amorphous salt. Found—Na_2O, 7·51; Sb_2O_5, 73·14; H_2O and loss, 19·35. Calculated—Na_2O, 7·18; Sb_2O_5, 74·06; H_2O, 18·74. At 100° it loses $4H_2O$.

In aqueous solution this salt splits up according to the identical reaction that has been given to the decomposition of the tetra-vanadates, into meta and trivanadates.

Tetra-antimonic acid was neutralised (to phthalein) with N/10 soda. The solution, left to itself, became acid, and a tribasic salt, $Sb_2O_5.Na_2O$, crystallised out. After the lapse of some days, the reaction—

$$6(Sb_2O_5)_2Na_2O = 3(Sb_2O_5)_3Na_2O + 3(Sb_2O_5.Na_2O),$$

being complete, heat was applied to dissolve the tribasic salt. This salt neutralises a part of the monobasic salt, the neutralisation is completed with N/10 soda. The acidity thus measured is found to be equal to one-third of the acidity of the original tetra-antimonic acid. It is thus $2Na_2O$. The reaction which expresses this neutrality—

$$3(Sb_2O_5)_3Na_2O + 3(Sb_2O_5.Na_2O) + 2Na_2O = 4(Sb_2O_5)_3(Na_2O_2),$$

agrees with the decomposition into an acid, and a tribasic salt; on account of this decomposition the tetra-antimoniate should be prepared with great rapidity.

Monobasic Salt, $(Sb_2O_5)_4.Na_2O$.—This salt has not been isolated. The reaction, not very distinct it is true, of tetra-antimonic acid on methyl-orange points to the existence of acid salts. The monobasic salt appears to be produced by the action of salts of sodium with mineral acids, on tetra-antimonic acid, with which sulphate, nitrate, and chloride of sodium in excess give a precipitate. The clear liquid does not contain any antimony, and the titration of its acidity shows that $1Sb_2O_5$ has set at liberty 1/2 HCl. This can only be explained by the formation of a salt of the formula $(Sb_2O_5)_4M_2O$. The precipitate is decomposed by washing.

Mixed Salt, $(Sb_2O_5)_4Na_2O.(Sb_2O_5)_2Na_2O.20H_2O$. —Products in the form of a crystalline powder, of which the composition is very similar to this, have been obtained by freezing: (1) A solution of $1Sb_2O_5$ and $3NaCl$; (2) a solution of $1Sb_2O_5$ and $\frac{1}{2}NaOH$. (1) Found—Na_2O, 5·93; Sb_2O_5, 77·72; H_2O and loss, 16·35. (2) Found—Na_2O, 5·35; Sb_2O_5, 78·52; H_2O and loss, 16·13. Calculated—Na_2O, 5·16; Sb_2O_5, 79·86; H_2O, 14·97.

Bibasic Tetra-antimoniate of Potash, $(Sb_2O_5)_2K_2O.9H_2O$. —Crystalline salt, found—K_2O, 10·87; Sb_2O_5, 70·42; H_2O and loss, 18·71. Amorphous salt, found—K_2O, 10·89; Sb_2O_5, 71·48; H_2O and loss, 17·63. Calculated—K_2O, 10·53; Sb_2O_5, 71·41; H_2O, 18·06. At 100° it loses $4H_2O$.

Bibasic Tetra-antimoniate of Baryta, $(Sb_2O_5)_2BaO.5H_2O$ (at 100°).—Found—BaO, 18·09; Sb_2O_5, 71·32; H_2O and loss, 10·59. Calculated—BaO, 17·36; Sb_2O_5, 72·45; H_2O, 10·19. It dissolves easily in concentrated HCl.

Monobasic Salt, $(Sb_2O_5)_4BaO,15H_2O$.—A crystalline powder obtained by freezing a solution of $1Sb_2O_5$ and $2BaCl_2$. Found—BaO, 8·42; Sb_2O_5, 75·43; H_2O and loss, 16·15. Calculated—BaO, 9·00; Sb_2O_5, 75·14; H_2O, 15·85. At 100° it loses $5H_2O$.

Action of Baryta on Tetra-antimonic Acid.—After several days contact of the two bodies in solution we obtain tribasic tri-antimoniate of baryta, $Sb_2O_5BaO,2H_2O$ (at 100°). Found—BaO, 29·35; Sb_2O_5, 60·92; loss on calcining, 7·91. Calculated—BaO, 30·11; Sb_2O_5, 62·82; H_2O, 7·07. This salt is slightly soluble in water, and easily so in HCl.

Bibasic Tetra-antimoniate of Copper, $(Sb_2O_5)_2CuO,9H_2O$. —Found—CuO, 9·12; Sb_2O_5, 72·48; H_2O and loss, 18·40. Calculated—CuO, 9·03; Sb_2O_5, 72·60; H_2O, 18·37. A green precipitate insoluble in water, soluble in tetra-antimonic acid. After several days contact with dilute potash it gives $Sb_2O_5K_2O$; the remaining precipitate, freed from the mother-liquor, is soluble in water. Tetra-antimoniate of copper dissolves slowly in ammonia, with which it forms an ammoniacal antimoniate, crystallising in hexagonal prisms.

Divers Tetra-antimoniates.—The silver salt is easily soluble warm. The salt of cadmium and that of mercury do not dissolve until after several days contact with water, without doubt after having undergone the same transformation as tetra-antimoniate of soda.

The salts of nickel and cobalt give ammoniacal antimoniates, blue for cobalt, red for nickel.

Triantimonic Acid.—For the preparation of this acid we used antimonic hydrate, well washed and dissolved hot.

The crystallised hydrate is prepared by the evaporation of the acid on the water-bath at 100°, or by spontaneous evaporation. That prepared by freezing, and dried in the air, is $Sb_2O_5,3H_2O$. Found—H_2O, 14·98. Calculated—H_2O, 14·44. Over sulphuric acid it becomes $Sb_2O_5,2H_2O$. At 100° it loses very little water. This crystallised hydrate answers to Geuther's amorphous product (*Bull. Soc. Chim.*, [3], xvii., 207).

The amorphous hydrate is difficultly soluble in concentrated hydrochloric acid. Water separates the tetra-antimonic hydrate from this solution.

Triantimoniates.—The preparation of the bibasic salts is analogous to that of the tetra-antimoniates. The alkaline salts are soluble in water, the solutions being opaline. The bi- and tri-basic salts are precipitated by alcohol, the first in the amorphous state, the others separate slowly in the crystalline state. The monobasic salts do not act in the same manner.

The salts of the other metals are insoluble in water, and rather soluble in tri-antimonic acid.

The bibasic tri-antimoniates are incompletely soluble in hydrochloric acid. They are distinguished from the tetra-antimoniates, inasmuch as, when treated by the alkaline bases, in the presence of water, they do not give tribasic triantimoniates, at least not at the ordinary temperature.

Bibasic Triantimoniate of Soda, $(Sb_2O_5)_3Na_2O,10H_2O$. —Amorphous salt found—Na_2O, 10·24; Sb_2O_5, 75·16; H_2O and loss, 14·60. Crystalline salt found—Na_2O, 10·14; Sb_2O_5, 75·37; H_2O and loss, 14·49. Calculated—Na_2O, 9·82; Sb_2O_3, 75·94; H_2O, 14·21. At 100° it loses $3H_2O$.

Monobasic Triantimoniate of Soda, $(Sb_2O_5)_3.Na_2O,11H_2O$. —The action of the salts of sodium with mineral acids on triantimonic acid is analogous to that of these reagents on the tetra acid.

With chloride of sodium $\frac{1}{3}HCl$ is set at liberty by $1Sb_2O_5$, and this is followed by the formation of an acid salt, $(Sb_2O_5)_3Na_2O$. Washing decomposes it. Freezing triantimonic acid, half neutralised by soda, gives a crystalline powder. Found—Na_2O, 5·15; Sb_2O_5, 78·81; H_2O and loss, 16·04. Calculated—Na_2O, 5·09; Sb_2O_5, 78·68; H_2O, 16·23. The action of triantimonic acid on methyl-orange, although not very distinct, also points to the formation of acid salts. To properly detect the end of the reaction requires very variable quantities of the bases, according to the alkali used.

To return to the yellow colour, only $\frac{1}{3}LiOH$ is required for $1Sb_2O_5$, while under the same conditions practically $\frac{1}{3}NaOH$ is required.

Bibasic Triantimoniate of Potash, $(Sb_2O_5)_3(K_2O)_2,10H_2O$. —The amorphous salt was prepared by precipitating with alcohol triantimonic acid treated with an excess of potash. Found—K_2O, 14·82; Sb_2O_5, 71·87; H_2O, 13·14. Calculated—K_2O, 14·20; Sb_2O_5, 72·26; H_2O, 13·54. Freezing gives only a mixture of the amorphous and crystallised salts. At 100° it loses $3H_2O$.

Bibasic Triantimoniate of Baryta, $(Sb_2O_5)_3BaO,5H_2O$ (at 100°).—A salt prepared by means of the acetate. Found—BaO, 22·72; Sb_2O_5, 69·98; H_2O and loss, 7·30. Calculated—BaO, 22·61; Sb_2O_5, 70·76; H_2O, 6·63. Insoluble in water, incompletely soluble in HCl, soluble in triantimonic acid.

9/10 *Antimoniate of Baryta*, $(Sb_2O_5)_{10}(BaO)_9,18H_2O$.— This salt results from the prolonged action of bartya water on the acid. Found—BaO, 28·62; Sb_2O_5, 64·81; H_2O and loss, 6·57. Calculated—BaO, 28·14; Sb_2O_5, 65·24; H_2O, 6·61. Besides its different composition this salt is

distinguished from the tribasic triantimoniate by its insolubility in HCl.

Bibasic Triantimoniate of Copper, $(Sb_2O_5)_3(CuO)_2 10H_2O$. —Found—CuO, 12·22; Sb_2O_5, 73·61; H_2O and loss, 14·17. Calculated—CuO, 12·25; Sb_2O_5, 73·89; H_2O, 13·86. When heated to dull redness it becomes almost black; at a higher temperature it decomposes with transient incandescence and emission of white antimonial fumes. It is insoluble in water, and soluble in triantimonic acid. Slightly soluble in NH_3, while the tribasic salt dissolves, giving a crystallised ammoniacal antimoniate of copper.

Bi-acid Triantimoniate of Copper, $(Sb_2O_5)_6CuO,16H_2O$. —This salt was prepared by exactly precipitating triantimonic acid by acetate of copper. Found—CuO, 3·11; Sb_2O_5, 84·10; H_2O and loss, 12·79. Calculated—CuO, 3·48; Sb_2O_5, 83·92; H_2O, 12·59. With dilute potash it gives a decided opaline solution.

I propose completing this research by the description of those antimoniates which have as yet only been mentioned, and of others I have prepared.

M. Sanderens, not noticing my previous research, claims to have obtained a new antimonic acid (*Bull. Soc. Chim.*, [3], xxi., p. 53). Such cannot be the case, for the solutions made, according to his description, must contain tri- and tetra-antimonic acids. This important fact has been overlooked by him.

As to the salts formed by these acids, we do not agree with regard to their composition. M. Sanderens attributes to them the general formula $Sb_2O_5M_2O$, which is different to the results described above.—*Bull. Soc. Chim.*, Series 3, xxi., No. 24.

NOTICES OF BOOKS.

A Text-book of Physiological Chemistry. By Olof Hammersten. Authorised Translation from the Author's Enlarged and Revised Fourth German Edition, by John A. Mandel. Third [American] Edition. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1900. Pp. 628, 8vo. Ill. Cloth.

The second American edition of this standard work was reviewed in the Chemical News, January 13th, 1899, and it is only necessary to add here that Professor Mandel, desirous of keeping his work up to date, has incorporated new matter from the fourth German edition.

Dr. Hammersten says in his Preface that this work does not claim to be a complete handbook, but only a concise text-book, and he has succeeded in condensing some portions by re-writing, so that this edition occupies fewer pages than the former.

The reviewer is reminded of Professor A. W. von Hofmann's "Einleitung in die Moderne Chemie," which bears on the title-page of the fifth edition the unusual words "*gekürzte und verbesserte Auflage*," thus recognising the important fact that condensation is compatible with improvement and brevity with excellence.

The Index to this book fills sixty-two columns.

Führer durch die gesammte Calciumcarbid- und Acetylen-Literatur. Bibliographie der auf diesen Gebieten bisher erschienen Bücher, Journale, Aufsätze in Zeitschriften, Abhandlungen und wichtigeren. Patentschriften, unter Mitwirkung von A[nton] Ludwig, herausgegeben von S. Calvary & Co. Berlin. 1899. 8vo. Pp. 51.

This Guide to the Literature of Calcium Carbide and of Acetylene contains nearly 1000 titles of books, pamphlets, and articles in periodicals relating to these subjects. The arrangement is alphabetical by authors, the typography is clear, and the matter shows care in editing. It includes the literature of apparatus, and cites papers in forty-six books and journals: it also refers to the more important American, German, British, and French patents. The bibliography was suggested by the Second International Acetylene Exhibition held at Budapest, and special attention is given to the technical side of the subject.

It is of interest to compare Dr. Ludwig's booklet with one published by the Smithsonian Institution in 1898—"Review and Bibliography of the Metallic Carbides," by J. A. Mathews. The two do not cover the same ground, but overlap in the section of calcium carbide: Dr. Ludwig treats of the industrial manufacture and applications of acetylene, Mr. Mathews of the metallic carbides in general regarded as chemical compounds; their treatment of the subject differs, yet each has its own peculiar advantages as well as its weaknesses. Mr. Mathews gives the dates of the journal-articles, Dr. Ludwig unfortunately omits them. On the other hand, the American omits initials of authors, and the German properly retains them; the former groups the references under specific headings, the latter adheres to the alphabetical plan; the German work has a commercial aspect, the American a purely scientific one. The two supplement each other. Those interested in acetylene would do well to secure both these pamphlets, which are sold at nominal prices, and further search in literature will probably become unnecessary.

H. C. B.

A Course of Practical Chemistry. By M. M. Pattison Muir, M.A. Part II. Intermediate. London: Longmans, Green, and Co. 1899. Pp. 234.

In this, the second part of "A Course of Practical Chemistry," the author has adhered to the plan adopted in the first part, viz., to give a course of laboratory work which will help to train the student in the methods of chemical inquiry.

The student has already gone through the experiments showing the principles of qualitative analysis in Part I.; the present volume deals principally with more advanced and complex qualitative analysis, and the ground work of quantitative analysis.

The book comprises five sections, dealing respectively with: I. Volumetric or titrimetric analysis. II. Preparation of certain compounds, and examination of some of their reactions. This section includes the preparation of a number of chromium pompounds, and experiments with nitric acid. III. Qualitative analysis of moderately complex mixtures of salts, also of metals and alloys, besides a number of appendices on the qualitative work. IV. The determination of some equivalent weights and some molecular weights. V. On gravimetric analysis. There are a few illustrations, but most of them could easily be dispensed with; students who have already gone through one year's course of practical chemistry, do not need an illustration of a wash-bottle, a bulb at the end of a glass-tube, how to pour a liquid into a filter, &c.

The book closes with a collection of tables, and a note on the application of the electrolytical ionisation theory to analytical reactions.

Artificial Perfumes ("Les Perfums Artificiels"). By Eugene Charabot, Professor of Chemical Analysis at the Commercial Institute of Paris. With 25 Figures in the Text. Paris; J. B. Baillière and Son. 1899. Pp. 296.

Of late years there has been a large amount of work done on perfumes; papers, often contradictory, have accumulated with extraordinary rapidity, while commercial competition has become more and more keen. It is therefore not without difficulty that M. Charabot has succeeded in picking out the most salient points, and discerning, among the many opinions expressed, which are the best substantiated by facts.

The present volume will be of great use to chemists

and others who are interested in the subject of preparing perfumes of a definite composition. Those products which are of the greatest interest from the point of view of their application are torpineol, vanilla, piperonal or heliotropine, ionone or artificial violet, and artificial musk. The author has also devoted a good deal of attention to several natural principles of definite composition, such as linalol, borneol, safrol, &c., which, though not directly used in the perfumery industry, serve as primary materials for the subsequent preparation of artificial perfumes.

The author is to be congratulated on having produced a useful practical work, and also on the table of contents and index, both of which appear to be very complete.

CORRESPONDENCE.

LAW OF ATOMIC WEIGHTS.

To the Editor of the Chemical News.

Sir,—Nobody has so powerfully claimed the attention of the scientific world to the mathematical laws of the atomic weights as you, Sir.

I will present you, therefore, the law that I have discovered and demonstrated to the Society of Naturalists in Warsaw the 15/27 January, 1900, as follows:—

I. The atomic weights disposed in a series from He to Na present the property that the sum of the members equidistant from the ends of the series is constant:—

He	+	F	=	4	+	19	=	23 = Na
Li	+	O	=	7	+	16	=	23 = Na
Be	+	N	=	9	+	14	=	23 = Na
B	+	C	=	11	+	12	=	23 = Na

II. The same law governs, approximately, the series of elements from He to K:—

He	+	Cl	=	4	+	35·45	=	39·45 = K	+0·34
Li	+	S	=	7·03	+	32·07	=	39·10 = K	−0·01
Be	+	P	=	9·08	+	31·02	=	40·10 = K	+0·90
B	+	Si	=	10·95	+	28·40	=	39·35 = K	+0·24
C	+	Al	=	12·01	+	27 11	=	39·12 = K	+0·01
N	+	Mg	=	14·04	+	24·28	=	38·32 = K	−0·79
O	+	Na	=	16	+	23·05	=	39·05 = K	−0·06
P	+	Ne	=	19·06	+	20·05	=	39·11 = K	

III. The same law governs, approximately, the series of elements from He to Cu:—

He	+	Co	=	4	+	58·93	=	62·93 = Cu	−0·67
Li	+	Ni	=	7·03	+	58·69	=	65·72 = Cu	+2·12 *
Li	+	Fe	=	7·03	+	56·02	=	63·04 = Cu	−0·36
Be	+	Mn	=	9·08	+	54·99	=	64·07 = Cu	+0·47
C	+	V	=	12·01	+	51·38	=	63·09 = Cu	−0·31
Ni	+	Ti	=	14·04	+	48·15	=	62·19 = Cu	−1·41 †
O	+	Ti	=	16	+	48·15	=	64·15 = Cu	+0·55
F	+	Sc	=	19·06	+	44·12	=	63·18 = Cu	−0·42
Na	+	Ca	=	23·05	+	40·07	=	63·12 = Cu	−0·48
Mg	+	K	=	24·28	+	39·11	=	63·39 = Cu	−0·21
Al	+	Ar	=	?					
Si	+	Cl	=	28·40	+	35·45	=	63·85 = Cu	+0·25
P	+	S	=	31·02	+	32·07	=	63·09 = Cu	−0·51

* Exception; due to triad Co, Ni, Fe.
† Exception.

I cannot attempt any experiments suggested by this law, because I am a mathematician, not a chemist.—I am, &c.,

Prof. Dr. N. Delaunay.

Russia, Varsovie,
Institut Polytechnique de l'Empereur Nicolas II.,
16/28 January, 1900.

LABORATORY LAMP.

To the Editor of the Chemical News.

Sir,—In reply to "Chemicus," I would recommend him to invest in a benzene lamp (to be obtained of Gustave Barthel, Leipzig, for about 16 shillings).

They deserve to be better known. A very high temperature is easily obtained and controlled (1 grm. coal, in a platinum crucible, can be burnt, in order to estimate the ash, in fifteen minutes). The lamp is easily kept in order, is quite safe to use, and my experience with it and the methylated spirit lamp is decidedly in favour of the former.—I am, &c.,

Rudolf E. Weissmüller.

Lukavac, Bosnia, January 22, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 3, January 15, 1900.

Numerical Laws of Chemical Equilibrium.—O. Boudouard.—There ought to exist a relation between the different factors of equilibrium (temperature, electromotive force, pressure, physical state, chemical nature, condensation, or concentration). This relation is such that each of the factors is a function, and a continuous function, of the others. This is known exactly in the law which concerns the correlative changes of pressure and temperature (Clapeyron's formula). There exists, further, at constant pressure and temperature, a certain relation between the proportions of different bodies present in a gaseous mixture in equilibrium (Isambert's experiments on ammonium sulphydrate). If L is the heat of the reaction at constant pressure; T, the absolute temperature; P, the total pressure of the mixture; $CC' \ldots C_1C_1'$, the concentrations of each of the bodies entering into the reaction; $nn' \ldots n_1n_1'$, the number of molecules of each of the bodies concerned in the reaction; N, the algebraical sum of $n+n' \ldots -n_1-n_1'$; the author arrives at the expression—

$$500\int L\,\frac{dT}{T^2} + N\log_e P + \log\frac{C^nC'^{n'}\ldots}{C_1^{n_1}C_1^{n_1'}} = \text{constant.}$$

The concordance of numbers found by this formula is satisfactory.

Electrolysis of Potassium Chloride.—A. Brochet.—Several theories have been brought forward to explain the presence of hypochlorites and chlorates after electrolysis of the chlorides of the alkalis and alkaline earths. The author studies the case of the electrolysis of a solution of sodium chloride, but he suppresses the reduction by the addition of a small quantity of potassium bichromate—a proceeding adopted by Muller. The results are shown on a curve. In the presence of potassium chromate, at a temperature of 20°, in a scarcely alkaline or neutral liquor, chlorate is formed, with a yield of 70 per cent. Without chromate, as Foerster has shown, the chlorate never exceeds 35 per cent; but it must be remembered that, in this latter case, half the quantity of electricity is employed in reducing the hypochlorite.

A New Crystalline Molybdenum Sulphide.—Marcel Guichard.—The author obtained, by the action of a very high temperature on molybdenum bisulphide, a crystalline sesquisulphide. This new sulphide can, at a red heat in sulphur vapour, be re-converted into the bisulphide. It is known that the bisulphide can otherwise be obtained by the action of a temperature below red heat

on the trisulphide. The bisulphide is the most stable in the neighbourhood of red heat. The sesquisulphide dissociates at a temperature approaching that at which it is formed, and yields metallic molybdenum. There is no molybdenum sulphide stable at a very high temperature. By starting with the trisulphide, MoS_3, we can obtain by successive dissociations at higher and higher temperatures, first the bisulphide (MoS_2), then the sesquisulphide (Mo_2S_3), and finally the metal.

Action of Magnesium on Saline Solutions.—Henri Mouraour.—The author continues his research on this subject, and finds that it is not only with the solutions previously examined that magnesium acts more vigorously than with water, but also with solutions of perfectly different salts, and especially with ammoniacal salts. It is probable that these solutions (chiefly the ammonium salts) act by dissolving the magnesia which, forming on the surface of the magnesium, would tend to arrest the action.

Journal de Pharmacie et de Chimie, Series 6, Vol. x., No. 9.

Analysis and Titration of Cacodylates.—H. Imbert and A. Astruc.—One of the authors has shown that cacodylic acid, $AsO\begin{matrix}OH\\CH_3\\CH_3\end{matrix}$, behaves as a neutral body to heliantine, and is monobasic to phenolphthalein; this leads to a very simple method of estimating this body. Since cacodylate of soda is theoretically neutral to phenolphthalein, and, on the other hand, cacodylic acid is neutral to heliantine, it appeared probable that the quantity of a monobasic acid, such as HCl, necessary to add to a sodic salt to make it neutral to heliantine, ought to be exactly one molecule of acid for one molecule of the salt, according to the equation $AsO\begin{matrix}ONa\\CH_3\\CH_3\end{matrix}+RH=AsO\begin{matrix}OH\\CH_3\\CH_3\end{matrix}+RNa$. By applying this reaction to several commercial samples, we obtained the following results:—

	I.	II.	III.
Quantity of pure dry cacodylate of soda, per cent	92·80	88·00	75·20

Neither of these samples was neutral to phthalein, but all had a distinct acidity. The most convenient method of working is the following:—Dissolve 1·6 grms. of cacodylate of soda in 100 c.c. of distilled water; 10 c.c. of this solution are taken, and made neutral to phenolphthalein. The solution, to which is added a few drops of heliantine, is then titrated with a solution of hydrochloric acid at 3·65 grms. per 1000, or sulphuric acid at 4·9 grms. per 1000. This solution is added with a graduated burette until methyl-orange indicates the termination of the reaction. If n is the number of c.c. of acid solution used, the sample under examination contains $n \times 10$ per cent of pure cacodylate of soda.

A New Method for the Gravimetric Estimation of Reducing Sugars.—Dr. Ph. Chapelle.—This method is based on the action of the centrifugal machine, which is now coming into more common use in laboratories; it is driven by water pressure from the main, and makes about 3000 revolutions per minute. 100 c.c. of Fehling's solution, containing 1 grm. of cuprous oxide, is used, and 25 c.c. of this solution is mixed with the sugar solution, and the volume made up to 37·5 c.c. The tube is heated in a bath of a solution of chloride of lime boiling at about 110° for six minutes in the case of glucose, and ten minutes for lactose. It is then put in the centrifugal machine. After two or three minutes, the precipitate adheres so strongly to the sides of the tube that the latter can be turned upside down and drained on linen. The deposit is taken up with boiling distilled water, and the tubes again submitted to centrifugal force, decanted, and dried at 150—180°. The whole operation does not take more than twenty-five to thirty minutes.

Analysis of the Gum of the "Grevillea robusta."—MM. Rœser and Puaux.—This gum is exuded on the branches and trunk of the tree; it has no smell, but an astringent taste. Dried at 100°, it takes an odour of caramel and becomes very friable. Its aqueous solution is colourless and limpid, or perhaps slightly clouded; it is neutral to litmus. It is easily precipitated by alcohol at 95°; it is also precipitated by the basic acetate of lead after the solution has been made slightly alkaline with ammonia. It reduces a cupro-potassic solution very slightly. It is not acted upon by silicate of potash, stannous chloride, or mercurous nitrate. When treated with mineral acids, this gum gives galactose and pentaglucose (arabinose); the quantity of the former being slightly in excess of that of the latter.

No. 10.

This number contains no matter of chemical interest.

No. 11.

New Process for the Titration of Iodide of Potassium.—E. Vincent.—The process devised by the author is based on the same principles as that of M. Berthet. It consists of reacting with iodic acid on iodine as follows:—$6HIO_3+5KI=5KIO_3+3I_2+3H_2O$. The five-sixths of iodine set free comes from the iodide; it is estimated by titration with hyposulphite of soda. One grm. of the iodide is dissolved in 1 litre of distilled water, and 2 grms. of iodic acid are dissolved in another litre of water. By means of a pipette, take 100 c.c. of each of these solutions and mix them together; measure 5 or 10 c.c. of hyposulphite solution containing 2·48 grms. per cent and containing 2 grms. of bicarbonate of potash per litre; run the iodine solution, drop by drop, from a burette into the hyposulphite solution until a faint yellow colour appears. This process is simple and rapid, and gives good results.

The Bubonic Plague.—M. Grimbert.—The bacillus of bubonic plague was discovered by Yersin in 1894, during the epidemic at Hong Kong. The disease occurs in four forms:—(1) Bubonic, (2) septicemic, (3) pneumonic, and (4) ambulatory; the latter form, which is very mild and sometimes overlooked, only occurs where the plague is endemic. The bacilli are met with in the blood, in ganglia, in phylactera, and in the pneumonic phlegm. The cocco bacillus of plague is non-motile, and forms chains when cultivated in broth; it does not liquefy gelatin, and does not give any characteristic culture on potatoes; it is aërobic, does not form spores, and is killed at a temperature of 50°; it produces a slightly diffusible toxin. It is of interest to note that the plague can be given to monkeys by inoculation, or through the trachea or nose, but never through the digestive organs.

MISCELLANEOUS

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th instant, Sir James Crichton-Browne, M.D., F.R.S., Treasurer and Vice-President, presiding. The Hon. Evelyn Ellis, the Hon. Everard Feilding, the Hon. Francis Robert Henley, Mr. C. E. Newton, and Mr. R. Oliver were elected members. The special thanks of the Members were returned to Mr. Charles Hawksley for his donation of £100, to Mr. Frank McClean for his donation of £25, and to Sir Andrew Noble, K.C.B., for his donation of £100 to the fund for the Promotion of Experimental Research at Low Temperatures. The Chairman reported the decease of Professor David Edward Hughes, F.R.S., a Manager of the Royal Institution, on January 22 last. The following resolution was submitted:—"Resolved,—That the Managers desire to express to Mrs. Hughes their heartfelt sympathy in her sad bereavement, and the deep regret of the Managers in losing by the death of Professor Hughes a most valued colleague." Professor Hughes

became a Member of the Royal Institution in 1882. He delivered a discourse on "Theory of Magnetism" (illustrated by experiments), on February 8, 1884. As a Manager and Vice-President he has rendered important services to the Institution. And his scientific researches, which have received world-wide recognition, have done much to promote the objects of the Institution.

Analysis of Sulphide of Antimony.—M. Kitzing.—1. *Total Sulphur.*—0·9 grm. of the sample, previously dried at 105—110°, is mixed with 5 grms. of tartaric acid, and dilute nitric acid added drop by drop; it is then concentrated until all action ceases. After standing for twenty-four hours a few c.c. of bromine are added, when it is again allowed to stand for a night; a few crystals of iodine are then added to facilitate the solution of the last traces of sulphur. The excess of bromine is driven off by heating on the water-bath and frequently adding water: it is then evaporated several times with hydrochloric acid to drive off the nitric acid. The residue is taken up with hydrochloric acid and tartaric acid diluted with boiling-water. After filtration the sulphur is precipitated with chloride of barium. 2. *Free Sulphur.*—Stibnite sometimes contains as much as 20 per cent of free sulphur. For the purpose of estimation, 10 grms. of the dry sample are exhausted with ether in a Soxhlet extractor. The exhaustion should last thirty hours. 3. *Antimony.*—0·2 grm. of the substance are dissolved as in 1, and filtered; the antimony is then precipitated with sulphuretted hydrogen. The precipitate is oxidised with fuming nitric acid, and the oxide of antimony, SbO_2, weighed. 4. *Water.*—Dry at 105—110° until the weight is constant. 5. *Matter Soluble in Water.*—Digest a few grms. of the sample in warm water on the water-bath; filter and evaporate the solution in a tared platinum crucible.—*Zeits. fur Offentl. Chem.*, iv., p. 830.

Titrimetric Estimation of Bismuth by Arsenious Acid in Alkaline Solution.—C. Reichard.—The process devised by the author depends on the reduction of bismuthic acid by arsenious acid in alkaline solution, and the estimation of the unused arsenious acid. The compound of bismuth is dissolved in an acid, and an excess of alkali is added to the solution thus obtained. After heating, a current of chlorine is passed through. The solution is boiled until the precipitate formed changes from an orange-red to an intensely deep red colour. The bismuthic acid is well washed with water. A solution of arsenious acid in caustic soda is then added, the solution containing 0·01 grm. As_2O_3 per c.c. The whole is then boiled until the red bismuthic acid is transformed into white hydroxide. It is then acidulated strongly with sulphuric acid and filtered hot. The arsenious acid which has not taken part in the reaction is estimated in the clear filtrate, by means of permanganate. It is advisable only to take small samples of bismuth.—*Zeit. Anal. Chem.*, 1899, xxxviii., p. 100.

Volumetric Estimation of Gold and Platinum.—Heinrich Petersen.—The method devised by the author is based on the following facts:—When we pour a solution of platinic chloride into a sufficiently concentrated and cold solution of potassic iodide, iodine is set at liberty, the amount of which can be estimated by means of hyposulphite of soda, The reaction takes place according to the following equations:—

(1). $PtCl_4+4KI=PtI_2+I_2+KCl.$
(2). $I_2+2Na_2S_2O_3=2NaI+Na_2S_4O_6.$

From this it results that 1 atom of platinum (194) is indicated by 2 molecules of sodic hyposulphite (316). With gold the reaction takes place in a similar manner:—

(1). $AuCl_3+3KI=AuI+2I+3KCl.$
(2). $AuI+Na_2S_2O_3=NaI+NaAuS_2O_3.$
(3). $I_2+2Na_2S_2O_3=NaI+2Na_2S_4O_6.$

3 molecules of $Na_2S_2O_3$ take part in the reaction, and it is only after the formation of the double salt $NaAuS_2O_3$ that the hyposulphite acts on the free iodine.—*Zeit. Anorg. Chem.*, 1899, xix., p. 59.

MEETINGS FOR THE WEEK.

MONDAY, 12th.—Society of Arts, 8. (Cantor Lectures). "The Nature and Yield of Metalliferous Deposits," by Bennett H. Brough.

TUESDAY, 13th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 14th.—Society of Arts, 8. "The Diffraction Process of Colour Photography," by Prof. R. W. Wood.

—— Institution of Mining and Metallurgy, 8. "The Gold-bearing Alluvial Deposits of the Klondyke District, by J. B. Tyrrell. F.G.S.

THURSDAY, 15th.—Royal Institution, 3. "Modern Astronomy," by Prof. H. H. Turner, M.A., F.R.S.

—— Chemical, 8. "Ammonium Amidosulphite" and "Products of Heating Ammonium Sulphites, Thiosulphates, and Trithionate," by Edward Divers and Masataka Ogawa. "Note on the Refraction and Magnetic Rotation of Hexamethylene," by S. Young, D.Sc., F.R.S., and Emily C. Fortey, B.Sc. "The Combination of Sulphur Dioxide and Oxygen," by Edward J. Russell and Norman Smith. "Note on the Estimation of Gases containing Sulphur," by E. J. Russell. "Apiin and Apigenin—II., Note on Vitexin" and "The Yellow Colouring Principles of Various Tannin Matters—VII.," by A. G. Perkin.

FRIDAY, 16th.—Royal Institution, 9. "Life in Indo-China," by H. Warington Smith, M.A., LL.M., F.R.G.S.

SATURDAY, 17th.—Royal Institution, 3. "The Idea of Tragedy in Ancient and in Modern Drama," by W. L. Courtney, M.A., LL.D.

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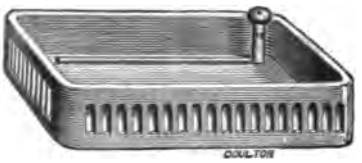

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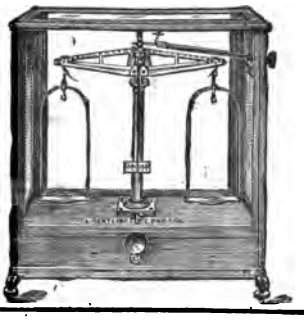

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ON THE

USE OF ICELAND SPAR AS A STANDARD IN VOLUMETRIC ANALYSIS.*

By ORME MASSON, M.A., D.Sc.

Introductory.

IN all volumetric work the first requisite is some substance of such a character that an exactly ascertained weight of it has a perfectly definite value in relation to the work in hand, whether this be alkalimetry, chlorine estimation, or what not. The strength of all the solutions employed is found by direct or indirect reference to this standard, and its selection is therefore a matter of fundamental importance. The substance employed must be readily obtainable in the required state; either chemically pure, in which case its value is calculable from its formula; or in pure aqueous solution, so that its value can be found by the measurement of a single physical property, such as density, or by the gravimetric estimation of a single chemical constituent. Where purity, either complete or partial is assumed, there should be no room for doubt as to the truth of the assumption. Where physical measurements or chemical estimations are necessary, the processes involved should be as simple as possible, not merely to save time and trouble, but more especially to minimise error. And, in any case, the standard substance should have no tendency to alter its composition on exposure to the air, as such a tendency renders troublesome precautions necessary, and even these do not always prevent error.

Few, if any, of the standards used in acidimetric and alkalimetric work approach the ideal. Oxalic acid is now generally condemned because of its variability in respect to hydration. The gravimetric analysis of solutions of hydrochloric or sulphuric acid by ordinary precipitation methods is not in much favour, nor is any process likely to be so which involves filtration, washing, and incineration. The analysis of sulphuric acid solution by converting it into the ammonium salt has been recommended, and is doubtless capable, like other methods, of giving correct results under certain conditions; but it is a serious objection that the salt to be weighed must be first obtained by evaporation, and then completely dried below 60° C. The fixing of the value of a sulphuric acid solution by determination of its density involves, in the first place, the assumption that the solution is as free from impurities as that employed by the investigator whose density tables are relied on, and, in the second place, it depends on a physical measurement which, without special precautions, cannot be trusted to give very accurate results. The only standard alkali in general use is sodium carbonate, which is made as required by heating a suitable quantity of pure bicarbonate. This method also fails of perfection; first, because the bicarbonate is liable to contain impurities (chlorides, sulphates, insoluble matter) and is then not at all easily purified; and, secondly, because the carbonate itself is a hygroscopic powder. It seems, then, that there is room for a standard of unquestionable and unalterable purity, and which can be used without any troublesome or risky manipulation.

Iceland spar is sometimes employed, as recommended by Fresenius, for verifying the standard of a hydrochloric acid solution, already determined by reference to sodium carbonate or otherwise. In this case excess of the acid is made to dissolve the whole of a weighed quantity of the spar, and the residual acid is then titrated by a previously standardised alkaline solution. Here, obviously, the Iceland spar is not itself an independent standard. It would become one, however, if so small an excess of acid were employed that no appreciable error could result from the use of a weak alkaline solution of only approximately known strength for the final titration. But this plan may be carried further; for it suggests employment of excess of the Iceland spar, with consequent complete neutralisation of the acid, and the calculation of the standard from the weight of spar dissolved. This method has been tried with encouraging results. The description of the procedure and the details of some test experiments may prove of interest.

Description of the Method.

An approximately normal hydrochloric acid solution is made in the usual way by the use of the hydrometer and tables of specific gravity. Compact fragments of Iceland spar, weighing from about 1 grm. to 3 grms. each, are broken from a larger crystal by pressing with the edge of a knife-blade and smartly tapping the knife with a hammer. These fragments are rinsed with dilute acid to free them from any adhering powder and to round their angles, well washed with distilled water, and dried at above 100° in an air-oven. They are kept for use in a stoppered bottle. One or more of these pieces, weighing altogether from 2½ to 3½ grms., are placed in a clean and dry beaker, and the whole is then accurately weighed. It is not necessary to know the exact separate weight of either beaker or spar. Suitable beakers are of about 20 grms. weight and 80 to 100 c.c. capacity. The beaker is then covered with a watch-glass, and exactly 20 c.c. of the hydrochloric acid are run in from a burette; the point of the burette, the spar in the beaker, and the cover, being so arranged that there can be no loss from the slight spirting that attends the first action. Small flasks may be used in place of beakers, but they are not quite so convenient in the subsequent operations. The covered beaker is then left till the evolution of gas has almost or quite ceased, which is the case in three or four hours. The cover and sides of the beaker are then rinsed down with a little distilled water, the cover replaced, and the beaker raised to the boiling-point and kept at—or about—that temperature for one hour. This drives off dissolved carbonic acid and ensures the completion of the action Some tendency to bump at the points of contact of the spar and the beaker is overcome by heating it on an asbestos card, with the flame regulated to a suitable size and placed—after boiling has begun—somewhat to one side. As an easy precaution against accident, the beaker may be fixed in position by means of a stout glass rod clamped horizontally and firmly across its cover. Under these conditions the operation hardly requires any watching. After the hour's heating, the perfectly clear and neutral calcium chloride solution is decanted off, and the beaker and its contained residual spar (still in compact lumps) are thoroughly washed with distilled water, dried at about 110°, cooled, and weighed. The difference between the original and final weighings gives the weight of Iceland spar dissolved by, and exactly equivalent to, the 20 c.c. of hydrochloric acid. It also gives, by direct reading and without calculation, the "factor" of the acid or the ratio of its true strength to that indicated by exact normality. For the molecular weight of calcium carbonate is, on the oxygen basis and as exactly as it can be given with present knowledge, 100 (compare the Report of the Committee of the German Chemical Society on Atomic Weights, 1898); and therefore 20 c.c. of a strictly normal acid should dissolve exactly 1 grm. If it is found to dissolve 1·0117 grms., it is 1·0117 times normal; if it dissolves 0·9907 grm., it is 0·9907 times normal; and so on. In practice, two or more such experiments should be conducted simultaneously, and the results may be considered

* Read at the Melbourne Meeting of the Australian Association for the Advancement of Science, January, 1900.

satisfactory if they do not differ by more than about one in a thousand. The residual spar may be returned to the stock bottle for future use.

Possible Sources of Error.

(1). The errors inseparable from a burette measurement need not be discussed. They include the errors of reading, of the burette graduation, and of temperature; but, of these, the second disappears, and the third is reduced to a minimum when readings are compared that are taken from the same part of the same burette and nearly simultaneously. With the burettes now constructed the error of reading is also very small.

(2). Loss of acid by spitting in the early stage and by bumping in the later stage—by which time, however, such loss could hardly be perceptible, as the acid is almost neutralised—is entirely avoided by ordinary precautions, as already described.

(3). Loss of particles of the residual spar during the decantation and washing does not, as a matter of fact, occur. The spar remains in compact pieces, the decanted solution is absolutely clear, and the washing is carried out with the utmost ease.

(4). Solution of a portion of the residual spar in the form of bicarbonate during the action in the cold might be expected to occur to some slight extent. If it did, the expulsion of the carbonic acid by boiling would be accompanied by the re-precipitation of normal carbonate; but not the slightest turbidity is observable.

(5). Incomplete neutralisation of the acid is conceivable, but does not occur if the operation is carried out as described. It has been proved that it does occur, even when boiling is continued for a long time, if but a small excess of spar is employed, *i. e.*, if too small a surface of action is given. A very small residual acidity was found in one case where 1½ grm. was used, the conditions being otherwise as usual; but with the usual amount of 2½ to 3½ grms. the action is always complete.

(6). Solution of some of the spar as normal carbonate, with a corresponding exaggeration of the standard of the acid, seems not unlikely. Fresenius states, on the authority of Hofmann and Weltzien, that the solubility of calcium carbonate in water, after prolonged boiling, is 1 in 28,500 parts, while conductivity methods have given it as about 1 in 77,000. If the small volume of liquid dealt with and the compact nature of the spar be borne in mind, it is evident that any error from this source greater than about 1 in 1000 (or 1 m.grm.) is unlikely; and in practice it is found to be not nearly so great. A very faint alkalinity is just observable in the decanted solution when it is boiled with litmus and compared with an equal quantity of distilled water and litmus, simultaneously boiled. As, however, this alkalinity is more than counterbalanced by a trace of the hydrochloric acid, and as it can be matched by a very few drops of centinormal potash (of which 40 drops would correspond to an error of 1 in 1000), it may safely be neglected.

(7). Action of the acid on the glass beaker need not be feared, as it is not heated till after the acid has been almost totally converted into calcium chloride.

(8). The same remark applies to any conceivable loss of hydrochloric acid by volatilisation.

Advantages of the Method.

(1). Iceland spar is probably as pure a substance as any that the chemist deals with. One of the best values for the atomic weight of calcium is that obtained by Erdmann and Marchand by calcination of the spar. Its purity is, moreover, quite independent of any exertion on the part of the chemist himself. Selected crystals may be bought for a few shillings a pound, and it forms part of the ordinary stock of a chemical laboratory. The other substance employed, hydrochloric acid, is easily obtained in a pure state; but the method does not at all depend on its purity, since only its acidity value is required.

(2). Only one fluid measurement from a burette and two weighings are involved in each determination, and the rest of the manipulation is of the simplest possible kind. The weighing of the materials (glass and Iceland spar) is not subject to the same errors as that of powders, such as sodium carbonate, or of liquids, such as dilute sulphuric acid. There is no transference of the spar from one vessel to another between the weighings.

(3). As already explained, the result is obtained without arithmetic except for the subtraction of one weighing from another. This is, of course, quite a minor advantage; but it possesses a certain fascination and some practical value.

Test Experiments.

The experiments epitomised in the following table were carried out in the order given and in the manner already described. Those bracketed together were done nearly simultaneously. The four tests of acid C were begun and finished in one day. Some of the others were begun in the evening and allowed to stand overnight before boiling.

In order to test the results by comparison of different experiments not only with the same acid, but with acids of different strengths of known relative value, the second acid was prepared from the first by diluting it in the exact ratio of 100 : 101, and the third acid was prepared in the same way from the second. The calculated and found strengths of these acids are seen to agree.

In order to test the results obtained by this method with those got by reference to other standards, two methods were employed. In the first place a normal solution of sodium carbonate was made in the usual manner. But the bicarbonate used in its preparation, though the best obtainable, was known to be not absolutely pure, as it contained traces of chlorine and insoluble matter; and the brief exposure of the carbonate, while its weight was being finally adjusted on the balance, would tend towards an error in the same direction. The solution might therefore be expected to prove slightly below, though certainly not much below, normal strength. Each sample of acid submitted to the spar test was also titrated with this sodium carbonate solution, using litmus and estimating the end point by direct comparison with distilled water and litmus, the test and the check being boiled simultaneously. The results are given in one column of the table in such a way as to show the strength of the acid on the (probably incorrect) hypothesis that the sodium carbonate was of exactly normal strength, and in another column in such form as to show the true strength of the sodium carbonate on the Iceland spar basis. This is seen to be 0·997 normal, as an average result.

In the second place, estimations were made of the chlorine strength of each sample of hydrochloric acid, except A, by titrating the calcium chloride obtained in the spar test by means of silver nitrate with the chromate indicator. The silver solution, which was approximately decinormal, was exactly standardised by a decinormal salt solution, made with specially purified and fused salt. The calcium chloride solution was made in each case by mixing the whole liquid and washings of a pair of tests and diluting to 500 c.c. This therefore contained the chlorine of 40 c.c. of the hydrochloric acid, and was approximately 0·08 normal. (The test for residual acidity or alkalinity, already referred to, was not applied till after this solution had been made up). The exact strength now found by the silver titration should agree with that given by the Iceland spar test, provided, in the first place, that both standards (sodium chloride and spar) are thoroughly trustworthy, and, in the second place, that the hydrochloric acid was pure in the sense that its acidity was precisely matched by its chlorine contents. No reason was known against either of these assumptions, and they seem justified by the results. These are given in two columns of the table so as to show, first, the strength of the acid on the sodium chloride basis, and, secondly, the strength of the sodium chloride solution on the Iceland spar basis. It is evident that silver solutions may be

Approximately normal HCl.	How made.	Calculated strength of the acid (normal=1).	Grms. of Iceland spar dissolved by 20 c.c. of the acid.	Mean result = strength of the acid by spar standard.	Strength of the acid by titration with "normal" Na_2CO_3.	Strength of the acid by titration with $AgNO_3$ (NaCl standard).	Strength of the Na_2CO_3 by the spar standard.	Strength of the NaCl by the spar standard ×10.
A.	Stock	(1·011)	1·0017 1·0107 1·0102 (see Note).	1·0109	1·014	—	0·997	—
B.	A diluted (100:101)	1·001	1·0005 1·0015	1·0010	1·005	1·003	0·996	0·998
C.	B diluted (100:101)	0·991	0·9907 0·9910 0·9913 0·9914	0·9911	0·993	0·991	0·998	1·000
D.	Mixture of acids, ratio not exactly ascertained. ..	—	1·0016 1·0015	1·0015	1·004	1·0015	0·998	1·000

standardised as correctly by indirect reference to Iceland spar as by direct reference to sodium chloride.

All titration values given, except the last one with sodium carbonate, are the means of two or more closely concordant results. The acid was always run from the same part of the same burette, and the other burettes employed were proved to agree with it to within less than 1 in 1000. The flasks employed were not directly compared with the burettes as to capacity; but any small error from this source could affect only the titrations—not the spar tests themselves, into which no flask measurement enters, nor the calculated dilution values to any appreciable extent. During the week in which the experiments were made, and even on single days, the laboratory temperature underwent rather considerable changes, which must account for a large part of the small differences observed, and may possibly explain also certain regularities which they show.

NOTE TO TABLE.—A fourth test of acid A was done simultaneously with the third, but differed from all others in that only about 1½ grms. of the spar were employed. This gave insufficient surface for complete action and caused residual acidity. Its amount was ascertained by titration with centinormal potash, of which 3·8 c.c. were required, corresponding to 0·0019 grm. of $CaCO_3$. The result read: Spar dissolved, 1·0085; add for residual acidity, 0·0019; acid standard, =1·0104. This was the only case of such a correction being required, and for that reason it is excluded from the table.

University of Melbourne.
January 1, 1900.

THE STUDY OF CARBON ELECTRODES.

By J. ZELLER.

THE author, after having pointed out the growing importance of the use of artificial carbon electrodes in the electro chemical industry, touches the question of their resistance to the action of electrolytes and of the products of electrolysis, and, in particular, of chlorine.

The author combats the opinion that the densest carbons would also last the longest. He specially quotes the observations made on the carbons made by Girard and Street, mark E. G., of the Society "Le Carbone," the density of which is 2, and which has a shorter duration than that of electrodes with less density. The same applies to Ceylon graphite.

According to the author the best point of comparison for the valuation of carbons used as electrodes would be the porosity, which is determined in the following manner:—

Let a be the true density of the carbon, taken on the powder, and b the apparent density. The porosity in terms of the true density is expressed as follows—

$$p = 100\,\frac{(a-b)}{a}.$$

If we admit with Winteler that the disaggregation of the carbons is principally due to the mechanical action of the gases, we should conclude that the less porous carbons are also the best.

Finally, we must take into account the relationship between the specific resistances of the carbon and of the liquid submitted to electrolysis. If the carbon has a higher resistance than the electrolyte, the density of the current in the electrodes would be greater in their upper portions, and consequently the disaggregation would be greater in this part, the result being that the carbons would have a tendency to break off at the level of the electrolyte. In the contrary case the carbons taper off, and become gradually thinner from the level of the liquid to the bottom. It is therefore necessary to make certain of having a uniform density of current at the surface of the electrodes, by proportioning as far as possible the two specific resistances of the electrodes and of the electrolyte. —*Zeits. fur. Elektr.*, 1899, vi., No. 40.

THE ESTIMATION OF IODIC ACID IN NITRATE OF SODA.

By R. AUZENAT.

IT is of importance to be able to exactly estimate the iodic acid present in raw nitrate of soda, certain samples containing as much as 1 or 2 per cent of iodate of soda.

The Beckurts method is rather qualitative, that of Rammelsberg is only applicable to the estimation of iodates in the presence of acids which have no action on iodide of potassium; however, by slightly modifying this latter method I succeeded in effecting the estimation in nitrates.

If to a solution of an iodate containing iodide of potassium, we add a small quantity of sulphuric acid, the iodic acid reacts on the iodide of potassium, setting iodine at liberty; agitation with a solvent, such as bisulphide of carbon or benzene, causes the iodine to pass into this medium, giving it a violet colour. In the presence of a nitrate, even when dilute, sulphuric acid sets free nitric acid, which decomposes the iodide of potassium; the perchlorate of potash which exists in the raw nitrates, acts in the same manner. If, on the contrary, we add a very small quantity of acetic acid to a dilute solution of the nitrate, the iodic acid reacts only on the iodide, and gives the liquid a caramel-yellow colour.

For its estimation I proceed in the following manner:—

In a test-tube rack, having the base covered with white paper, I placed six test-tubes of the same size, 25 m.m. diameter and 18 c.m. high; a mark on each shows the level of 50 c.c. In the first I place 10 c.c. of a solution of iodate of potassium containing 1 grm. per litre; in the second, 30 c.c of a solution of the raw nitrate containing 33 grms. per litre; in the third, 35; in the fourth, 40; in the fifth, 45; and in the sixth, 50 c.c. To each of the first five tubes I add distilled water up to the mark, then to all six 2 c.c. of a solution of iodide of potassium at 10 per cent, and, as rapidly as possible, five drops of glacial acetic acid.

The caramel colour immediately appears and becomes homogeneous after a slight agitation. At the end of ten minutes we note which tube corresponds with the colour of the standard tube. If the five tubes are all darker coloured than the standard tube, we repeat the experiment with 20 c.c., or even more, of the titrated solution of iodate; if the five tubes are less deeply coloured than the other one, we, on the contrary, reduce the amount of iodate.

It is indispensable to fill all the tubes afresh for each experiment, as the colour changes with the time, and the comparison could not otherwise be made under the same conditions as to time.

Thus, with tubes of a lighter colour than the standard, the titration would be impossible by the addition of the typical solution to one of them, until the colours were identical.

Under the conditions shown above, the estimation can be made with the same rapidity, and the same exactitude as the estimation of ammonia with Nessler's solution.—*Moniteur Scientifique*, Series 4, xiv., February, 1900.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART II.

TUNGSTEN AND MOLYBDENUM.

By HARRY BREARLEY.

(Concluded from p. 65).

MOLYBDENUM.

I. Separation from Other Elements

275. *Iron.*—Gentele (*C. N.*, iii., 223).—Molybdates of iron are formed when neutral $(NH_4)_2MoO_4$ is added to a ferric solution. (Such combinations prevent the perfect separation of Mo from Fe, Cr, and Al by ammonia, &c.—H. B.).

275*a*. Bamber (*J. I. S. I.*, 1894, i., 319).—Dissolve steel in HNO_3, add Na_2CO_3, but not to alkalinity, evaporate, ignite to dull redness, and digest with dilute Na_2CO_3. Mo, P, S, Cr, and Va are dissolved out. Mo and Va generally occur in basic pig containing Cr.

276. *Phosphorus.*—Debray (*C. N.*, xvii., 183).—The phospho-molybdic acid, mixed with lime, is heated in successive currents of H_2S and HCl. Wash with water and with HCl, and filter off molybdenum sulphide.

277. Reichardt (*C. N.*, xxviii., 315).—Dissolve in sodium carbonate, and precipitate P with magnesia mixture.

278. *Chromium.*—Benneville (*J. I. S. I.*, 1895, i., 206).—Chromium precipitated with excess of ammonia; operation needs repeating (see 281*a*).

279. *Tin.*—Clarke (*C. N.*, xxi., 124).—Metals precipitated as sulphides, and the stannic compound redissolved by a faint HCl solution of oxalic acid. W behaves erratically.

280. *Arsenic.*—Friedheim and Michaelis (*J. C. S.*, lxviii., 415).—Arsenic is distilled after mixing with methylic alcohol and HCl. The separation from tungsten is less perfect; they may, however, be precipitated together with $HgNO_3$, and the As estimated by difference.

281. *Vanadium.*—Carnot (*J. C. S.*, lii., 897).—Va is precipitated from ammoniacal solution containing $(NH_4)Cl$ as manganese vanadate. Like separation of W not complete. (See also 298).

281*a*. *Various.*—Brearley (*C. N.*, lxxix., 2).—Ba is precipitated with H_2SO_4 from HCl solution. By precipitating as $PbMoO_4$ in modified ways Ba, Ca, Sr, As, Cd, P, Al, Ur, and those elements named already (283) do not interfere. Silicates give slightly high results. Fe and Cr are separated at boiling-point with soda hydrate. Separation of Cr with ammonia (278) not good.

II. Gravimetric Estimations.

282. Chatard (*C. N.*, xxiv., 175).—Mo precipitated with lead acetate, ignited apart from filter, and weighed as $PbMoO_4$. Analogous process for tungsten frustrated by fineness of the precipitate. (See 243).

283. Brearley (*C. N.*, lxxviii., 203).—Critical examination of Chatard's process. $PbMoO_4$ may be ignited with the paper. Prolonged heating at high temperature causes only slight loss. Presence of excess acetic acid, lead acetate, alkaline nitrates, chlorides, or acetates, and salts of the metals Mn, Cu, Co, Ni, Zn, Mg, Hg are without influence. Schindler's volumetric process (295) supplemented by a more delicate end-reaction.

284. Von der Pfordten (*C. N.*, l., 18).—Precipitation with $HgNO_3$ and reduction in hydrogen.

285. Jannasch and Wasowicz (*C. N.*, lxvi., 6).—Similar to 284.

286. Moissan (*C. N.*, lxxii., 2).—Precipitates as mercurous molybdate, and determines as bioxide. (This admirable paper gives an account of the preparation and numerous properties of metallic Mo.—H. B.).

287. Smith and Bradbury (*J. C. S.*, lxii., 241).—Same available precipitants as for W (see 261). Suggest the estimation of W and Mo together as Cd salts and electro-estimation of Cd in another portion as a means of determining W and Mo in mixtures.

288. Pechard (*C. N.*, lxv., 89).—Volatilised in HCl gas as $MoO_3.2HCl$. The compound is dissolved in water, evaporated, and MoO_3 weighed. Tungstates are not at all volatilised, and may be thus separated (234 and 235).

289. Smith and Hoskinson (*C. N.*, liii., 278).—Deposited electrolytically from acid solution of the molybdates as $Mo_2O_3.3H_2O$ and so weighed, or ignited to MoO_3.

289*a*. Heidenreich (*J. S. C. I.*, 1896, 744).—Mo_2O_3 is not completely deposited from solution of $(NH_4)2MoO_4$ in four days.

290. Friedheim and Euler (*J. C. S.*, lxviii., 535).—Precipitated as sulphide, filter, ignited short of losing Mo, digested with $(NH_4)HO$, evaporated, and ignited to MoO_3.

291. Taggart and Smith (*J. C. S.*, lxxii., 433).—Mo sulphide ignited with oxalic acid burns to pure MoO_3.

III. Volumetric Estimations.

292. Werncke (*C. N.*, xxxii., 259).—In Pisani's process (reduction with Zn and titration with permanganate) the reduction is not to the sesquioxide. Sodium amalgam does not effect a complete reduction. The results are mutually comparable.

293. Benneville (*J. I. S. I.*, 1895, i., 228).—Pisani's process. The temperature at which the reduction is made has no influence on results; nitric acid must be absent; H boiled off before titrating.

294. Von der Pfordten (*C. N.*, l., 18).—HCl solution reduced and titrated in presence of H_2SO_4 and $MnSO_4$. Similar process for W; the reduced solu-

tion (red) added to excess permanganate; excess estimated with $FeSO_4$.

295. SCHINDLER (*C. N.*, lviii., 61).—Faintly acetic solution precipitated with standard lead acetate, using tannin as indicator. Lead may be estimated inversely (see 283).

296. SEUBERT and POLLARD (*C. N.*, lxxii., 293).—MoO_3 is dissolved in standard soda and excess titrated with HCl and phenolphthalein.

297. FRIEDHEIM (*C. N.*, lxxv., 91).—Molybdate decomposed with KI and HCl; iodine absorbed and titrated with thiosulphate. A discussion of modified process by Gooch and Fairbanks. Gooch defends his modifications (*C. N.*, lxxv., 208; and *C. N.*, lxxviii., 207).

298. FRIEDHEIM and EULER (*J. C. S.*, lxviii., 535).—By distilling first with HCl and HBr, and then with HCl, KI, and phosphoric acid, both Va and Mo are estimated from the Br and I liberated.

IV. DETECTION AND MISCELLANEOUS NOTES.

299. SCHLOSBERGER (*C. N.*, iii., 192 and 204).—Dilute solution of $(NH_4)_2MoO_4$ gives blue colour with a soluble sulphide.

300. SKEY (*C. N.*, xvi., 324).—An HCl solution shaken with zinc gives a red colour, which detects one part in two millions. Fe does not interfere if time be allowed it to deoxidise. W gives a like colour. (See also ELBRAM, *J. C. S.*, lxxii., 522).

301. MOISSAN (*C. N.*, lxxii., 2).—Heated with phosphorus perchloride, reddish fumes condense in a ring.

302. SCHONN (*C. N.*, xxi., 166).—Heated with H_2SO_4 a blue colour appears. MASCHKE (*C. N.*, xxxviii., 22) describes a more convenient and certain modification of the test.

303. SMITH (*J. C. S.*, lxiv., 171).—Mo precipitates Ag from ammoniacal AgCl quantitatively; similarly from solutions of auric chloride; tungsten acts in a similar way; Mo and W (less readily) reduce HgCl. (Would these reactions serve to measure the oxidation of metallic W and Mo powders?—H. B.). MoO_2 also precipitates Ag. (See SMITH and SHINN, *C. N.*, lxx., 197; also No. 264).

304. MONTEMARTINI (*J. C. S.*, lxii., 1402).—Strong HNO_3 attacks Mo slowly; more dilute acids do not at once convert what they dissolve into MoO_3.

305. GUICHARD (*J. C. S.*, lxxii., 496).—Mo alone is formed on igniting MoO_3 in hydrogen at 600° C.

306. IGELSTRÖM (*J. C. S.*, lxviii., ii., 505).—In Swedish hæmatite Mo_2O_3 was found up to 9 per cent.

ON GADOLINIUM.*

By C. BENEDICKS.

(Concluded from p. 63).

Gadolinium Nitrate.—1. $Gd(NO_3)_3+6\frac{1}{2}H_2O$, is obtained as large soluble asymmetrical crystals, over sulphuric acid, or by free evaporation in air.

Analysis.

	I.	II.		
Gd ..	33·97	33·80 per cent.	Calculated	33·99 p. c.
$3NO_3$.	—	—	„	40·52 „
$6\frac{1}{2}H_2O$.	—	—	„	25·49 „

Mean specific gravity .. .. .. 2·332
Molecular volume .. .. .. 196·8

2. $Gd(NO_3)_3+5H_2O$, crystallises from concentrated nitric acid in beautiful monosymmetric prisms, which gradually become moist in air, and therefore are not suited to measurement.

* *Zeitschrift für Anorganische Chemie*, xxii., 5, p. 393.

Analysis.

	I.	II.	III.		
Gd ..	35·85	35·95 p. c.	—	Calculated	36·11 p. c.
$3NO_3$	—	—	42·81 p. c.	„	43·06 „
$5H_2O$	—	—	— „	„	20·83 „

Mean specific gravity .. .. 2·406
Molecular volume .. .. .. 179·5

Gadolinium Ammonium Nitrate was obtained as long, hair-fine, deliquescent needles, from a solution almost solidified over H_2SO_4, containing the respective nitrates in the proportion $1Gd.3(NO_3):2NH_4NO_3$. This falls in with the idea that gadolinium remains in the first mother-liquors with the yttrium metals, on the ammonium double nitrate crystallisations, whilst lanthanum, cerium, praseodymium, &c., crystallise out.

Gadolinium Sulphate, $Gd_2(SO_4)_3+8H_2O$, was obtained as small sparkling crystals at the ordinary temperature or on the water-bath. Crystalline form monoclinic, isomorphic with yttrium sulphate, $Y_2(SO_4)_3+8H_2O$.

Analysis.

	I.	II.	III.		
Gd_2O_3	—	—	—	Calculated	48·39 p. c.
$3SO_3$.	—	—	—	„	32·26 „
$8H_2O$.	19·51	19·46	19·64 p. c.	„	19·35 „

Specific gravity .. .. 3·010
Molecular volume .. .. .. 247·2

Anhydrous Gadolinium Sulphate, $Gd_2.3(SO_4)$.

Specific gravity .. .. 4·139
Molecular volume .. .. .. 145·0

Solubility determinations of the sulphate were conducted by dissolving small portions of the anhydrous salt by stirring in water, and then shaking the solution four to five hours, automatically, in a water-bath at the required temperatures. The graphic representation of the results shows that the solubility is much greater at 9·3° to 10·6° than at higher temperatures, the numbers obtained probably bearing a relation to the presence of a sulphate containing varying amounts of water.

Gadolinium Potassium Sulphate,—

$$Gd_2.3(SO_4)+K_2SO_4+2H_2O,$$

was obtained as large crystals on the water-bath from a solution containing $Gd_2.3(SO_4)+3K_2SO_4$.

Analysis.

Gd_2O_3 ..	44·49 per cent.	Calculated	44·43 per cent.
$3SO_3$ ..	—	„	29·62 „
K_2SO_4 ..	21·77 „	„	21·51 „
$2H_2O$ ..	4·39 „	„	4·44 „

Specific gravity .. .. 3·503
Molecular volume .. .. .. 231·4

Solubility in Saturated K_2SO_4 Solution.—According to my determinations, 100 c.c. saturated K_2SO_4 solution contains 0·87—0·77 grm. Gd_2O_3; according to Bettendorff, as the mean of three experiments, 0·77 grm. Gd_2O_3.

Gadolinium Selenate, $Gd_2(SeO_4)_3+10H_2O$; crystallises over sulphuric acid at the ordinary temperature in large clear prisms. The salt is isomorphous with the selenates of yttrium and erbium, whose formula given by Cleve is $R_2(SeO_4)_3+9H_2O$. A water content of $10H_2O$ agrees better, however, with his analysis results.

This salt decomposes rapidly, especially in the warm, wherefore crystallographic measurement is rather difficult. The crystalline form is rhombic.

Analysis of Salt.

	I.	II.	III.			
Gd_2O_3	38·95	38·82	—	p. c.	Calculated	39·57 p. c.
$3SeO_3$	—	—	—	„	„	41·39 „
$10H_2O$	[20·67]	[20·48]	20·11	„	„	19·54 „

Specific gravity .. .. 3·048
Molecular volume .. .. .. 302·3

2. $Gd_2.3(SeO_4)+8H_2O$ was obtained on the water-bath as large beautiful crystals, with somewhat the sheen of mother-of-pearl.

The salt is isomorphic with yttrium sulphate, $Y_2.3(SO_4)+8H_2O$.

Analysis.

	I.	II.		
Gd_2O_3	—	—	Calculated	40·66 per cent.
$3SeO_3$	—	—	,,	43·07 ,,
$8H_2O$	16·18	16·25 p. c.	,,	16·27 ,,

Specific gravity .. .. 3·309
Molecular volume .. .. .. 267·5

Anhydrous Gadolinium Selenate, $Gd_23(SeO_4)$, is obtained by heating the above selenate to 130°.

Analysis.

	I.	II.		
Gd_2O_3	47·76	47·80 per cent.	Calculated	48·56 p. c.
$3SeO_3$	[50·15]	51·61 ,,	,,	51·44 ,,

Specific gravity .. .. 4·175
Molecular volume .. .. .. 177·5

Gadolinium Potassium Selenate,—

$$Gd_{33}(SeO_4)+3K_2SeO_4+4H_2O,$$

was obtained on the water-bath as microscopically small needles.

Analysis.

Gd_2O_3..	24·99 per cent.	Calculated	24·36 per cent.
$6SeO_3$..	50·95 ,,	,,	51·61 ,,
$3K_2O$ ..	—	,,	19·15 ,,
$4H_2O$ ..	4·71 ,,	,,	4·88 ,,

Acid Gadolinium Selenite, $Gd_23(SeO_3)+H_2SeO_3+6H_2O$, was obtained by precipitating a solution of $Gd.3(CH_3CO_2)$ with $Na_2SeO_3+H_2SeO_3$, when an amorphous precipitate was formed which gradually passed into small needles.

Analysis.

	I.	II.		
Gd_2O_3..	—	38·94 per cent.	Calculated	38·69 p. c.
$4SeO_2$..	48·05	—	,,	47·77 ,,
$2H_2O$ ..	—	—	,,	3·87 ,,
$5H_2O$ ..	9·46	—	,,	9·67 ,,

Gadolinium Hyposulphate.—From a solution obtained from gadolinium sulphate and barium hyposulphate, little crystals deposited themselves which proved to be gadolinium hyposulphate. The solution solidified, after prolonged concentration over H_2SO_4, to a crystalline mass, very deliquescent, and incapable of analysis.

Gadolinium-ethyl Sulphate, $Gd.3(C_2H_5SO_4)+9H_2O$, obtained by double decomposition of gadolinium sulphate and barium-ethyl sulphate. The crystals are transparent rounded plates.

Analysis.

	I.	II.		
Gd	22·57	22·59 p. c.	Calculated	22·51 p. c.
$3C_2H_5SO_4$.	—	—	,,	54·11 ,,
$9H_2O$.. ..	—	—	,,	23·38 ,,

Specific gravity .. .. 1·923
Molecular volume .. .. .. 360·4

Gadolinium Vanadate. $Gd_2O_3.5(V_2O_5)+26H_2O$, obtained by precipitating a neutral gadolinium nitrate solution with a concentrated solution of sodium bivanadate and filtering off the precipitate. Crystals soon form in the filtrate.

Analysis.

Gd_2O_3 ..	21·02 per cent.	Calculated	20·69 per cent.
$5V_2O_5$ ..	52·04 ,,	,,	52·41 ,,
$26H_2O$ ..	26·93 ,,	,,	26·90 ,,

Specific gravity .. .. 2·660
Molecular volume .. .. .. 654·1

Gadolinium Carbonate.

1. *Basic Carbonate*, $Gd\begin{cases}OH\\CO_3\end{cases}+H_2O$ (dried at 100°).

A crystalline carbonate is obtained by passing CO_2 into cold water in which gadolinium hydroxide is suspended.

Analysis.

	I.	II.		
Gd_2O_3 ..	72·21	72·22 p. c.	Calculated	72·58 p.c.
$2CO_2$ ..	17·85	17·07 ,,	,,	17·74 ,,
$3H_2O$ ..	—	—	,,	9·68 ,,

2. *Neutral Carbonate*, $Gd_2.3(CO_3)+13H_2O(?)$. If CO_2 is passed in for a still longer time, it is observable through the microscope that the fine needles of the basic carbonate pass into larger oval crystals. It was not possible to get a constant weight, even after drying these crystals at 100° for three days.

Analysis.

	I.	II.		
Gd_2O_3 ..	49·35	49·66 p. c.	Calculated	49·59 p. c.
$3CO_2$ ..	—	[15·74] ,,	,,	18·18 ,,
$13H_2O$ ..	—	—	,,	32·23 ,,

Gadolinium Oxalate, $Gd_2.3(C_2O_4)+10H_2O$, a fine crystalline precipitate, which can be obtained from concentrated HNO_3 as large monoclinic crystals. On heating to 110° the salt loses $6H_2O$.

Analysis of Salt, dried in Air.

Gd_2O_3 ..	47·33 per cent.	Calculated	47·62 per cent.
$3C_2O_3$ ..	—	,,	28·57 ,,
$10H_2O$ ..	—	,,	23·81 ,,

According to Brauner, gadolinium should stand between neodymium and praseodymium, judging by his results for the solubilities of the three corresponding oxalates in 100 c.c. normal sulphuric acid, and which are respectively 0·10034 grm., 0·1095 grm., and 0·12327 grm.; therefore, gadolinium oxalate belongs to the oxalates of the rare earths most insoluble in acids.

Gadolinium Acetate, $Gd.3(C_2H_3O_2)+4H_2O$, obtained from the solution of the oxide in acetic acid, as difficultly soluble, clear, asymmetric crystals, isomorphous with the corresponding yttrium compound.

Analysis.

	I.	II.		
Gd_2O_3 ..	44·5	44·64 p. c.	Calculated	44·44 p. c.
$3C_4H_6O_3$.	—	—	,,	37·78 ,,
$8H_2O$ ..	18·09	— ,,	,,	17·78 ,,

Specific gravity .. .. 1·611
Molecular volume .. .. .. 251·4

Gadolinium Propionate $Gd.3(C_3H_5O_2)+3H_2O$, obtained as large rhombic crystals in the vacuum desiccator.

Analysis.

	I.	II.		
Gd_2O_3 ..	41·96	42·04 p. c.	Calculated	41·96 p. c.
$3C_6H_{10}O_3$	—	—	,,	45·45 ,,
$6H_2O$ ..	12·65	12·41 ,,	,,	12·59 ,,

Conclusions.

From these experiments it is evident that gadolinium is closely allied to the yttrium metals, and resembles the cerium metals to a much slighter degree. Therefore a small intermediate group is formed of samarium and gadolinium,

For trivalent elements, most characteristic are the platinochloride and gold chloride double salts, the potassium double sulphate, the selenite, the ethyl sulphate, and the basic carbonate of gadolinium.

Nothing can yet be definitely stated with regard to the position of gadolinium in the periodic system, but it will certainly find place in the eighth horizontal row.

PREPARATION OF GRAPHITOIDAL SILICON.*

By FREDERICK S. HYDE.

THE analogy between silicon and carbon in their allotropic modifications, especially the graphitic, and the existence of a crystalline compound of silicon and carbon, known as carborundum, have tended to create doubt as to the purity of graphitoidal silicon, yet the production of the latter seems to be based on reactions favouring the exclusion of carbon.

The usual methods involve the reduction of potassium-silico-fluoride with aluminum or metallic sodium; or the reduction of pulverised white sand with magnesium powder.

The method submitted depends on the reduction of finely pulverised white sand with magnesium powder and the subsequent fusion of the reduction-product with cryolite and aluminum, to form an alloy of aluminum and silicon, from which the latter is obtained on treatment with hydrochloric acid.

For experiment take :—

	Grms.
Finely pulverised white sand..	12
Magnesium powder..	3

Mix thoroughly, place in a dry test-tube, supported by a clamp on a ring stand, warm gently, and then commencing at the lower end ignite cautiously with the burner in hand.

There may be a slight separation of the contents due to the expansion of air or moisture, which should be remedied. As the heat becomes concentrated at the lower end, the contents become darker, resulting in a quiet incandescence which creeps toward the upper part, usually completing the reaction with a slight puff. The exterior surface of the test-tube assumes a greenish black lustrous appearance, and becomes wrinkled or "buckled" from the heat of the reaction. Remove the contents, which are brownish-black in colour; pulverise and mix with a sufficient quantity of cryolite powder.

Introduce the mixture into a small Denver crucible, containing a piece of aluminum (walnut size) on the bottom. The cryolite serves as a vehicle for incorporating the reduction product with the aluminum and as a flux for impurities. Place the crucible in a small gas furnace and apply blast.

As soon as the aluminum liquefies, stir the mass thoroughly with an iron rod. Quite a little stirring is necessary in order that the molten aluminum may be brought into contact with every portion of the reduced mass. Near the final stage, fumes and tongues of bluish flame, like burning zinc, may be observed on the surface. Allow to cool.

Remove the regulus, which should have a brilliant silver-white crystalline surface, and examine the blackened slag for any small globules of alloy. Place the regulus and globules in a porcelain dish or casserole, and treat with warm hydrochloric acid. As the aluminum dissolves, black glistening spangles of silicon separate out of the solution. Decant and wash with clear water. Then wash with alcohol and dry on the filter.

Some employ zinc to form the alloy, but aluminum is preferable for the following reasons not stated in the text-books :—

First.—Zinc melts and volatilises at a comparatively low temperature, before the silicon has a chance to become properly alloyed. It is also difficult to combine the reaction-product with molten zinc, since the latter is heavier and causes the former to float.

Second.—The copious white fumes and blue flame evolved by zinc, even though the crucible be covered and the oxidation minimised, cause large amounts of "zinc wool" to condense on the interior of furnace and crucible, thus interfering with the operation.

Third.—The zinc-silicon alloy is hard and resists solution with hot or cold hydrochloric acid, while the aluminum alloy is readily dissolved.

Fourth.—Zinc tends to produce needle-shaped crystals of silicon, which are not so striking as the graphitoidal spangles obtained from the aluminum alloy.

Graphitoidal silicon resists oxidation before the blow-pipe. Under the microscope, by reflected light, the faces of incomplete octahedra are visible in the small irregularly shaped masses. The yield is rarely more than 10 per cent by weight of the silica employed.

There are indications that the element silicon itself, heretofore classed among laboratory curiosities, may become commercially important.

On account of its high electrical resistance, its use in in the form of compact rods has already been suggested for electric heating apparatus.

* Contributions from the Havemeyer Laboratories of Columbia University. From the *American Chemical Journal*, vol. xxi., No. 8.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 1st, 1899.

Professor THORPE, F.R.S., President, in the Chair.

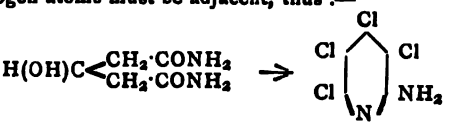
CERTIFICATES were read for the first time in favour of Messrs. Frank Curzon Britten, 21, Osbaldeston Road, London, N.; Robert Eadie, junr., 31, Clarendon Street, Partick, N.B.; Morris Broad Fowler, 30, West Park, Clifton, Bristol; Reginald Gordon Halstead, 3, Rathbone Place, Oxford Street, London, W.; James Henry Kershaw, 21, Richmond Park Road, Kingston-on-Thames; Thomas Macara, 6, West Bank Terrace, Hillhead, Glasgow; James Walter Tilley, 2, Stockwell Crescent, London, S.W.; Augustus John Walker, 3, Arnold Street, Hull.

Of the following papers, those marked * were read:—

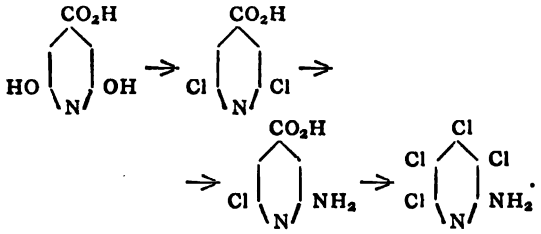
*8. "*The Chlorine Derivatives of Pyridine.* Part V. *Constitution of Citrazinic Acid. Formation of aa'-Dichloropyridine and of aa'-Diiodoisonicotinic Acid.*" By W. J. SELL, M.A., and F. W. DOOTSON, M.A.

Although its genetic relationships leave little room for doubt that in citrazinic acid the hydroxyl groups occupy the *aa'*-positions relatively to the nitrogen, in view of the importance of this compound and its derivatives it was deemed expedient to obtain confirmatory evidence. This was done as follows :—

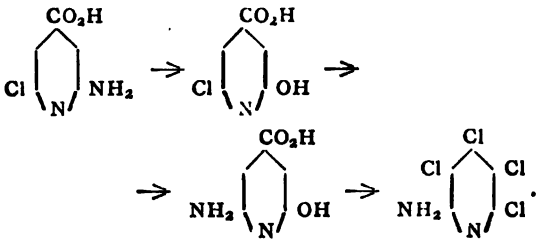
The diamide of *β*-oxyglutaric acid was prepared and treated with phosphorus pentachloride, when aminotetrachloropyridine was obtained. In this compound, the nitrogen atoms must be adjacent, thus :—



By the action of phosphorus pentachloride on citrazinic acid, dichloroisonicotinic acid was obtained by Behrmann and Hofmann (*Ber.*, 1884, xvii., 2694), and by the authors (*Trans.*, 1897, lxxi., 1069), and this interacts with ammonia to form aminochloroisonicotinic acid. (*Trans.*, 1897, lxxi., 1075), which substance, on further treatment with phosphorus pentachloride, loses its carboxyl group, and yields an aminotetrachloropyridine identical with that mentioned above. These changes may be represented thus :—



In this way, the position of one of the hydroxyl groups is shown. That both hydroxyl groups occupy the same relative position in the molecule is demonstrated by the following reactions: Aminochloroisonicotinic acid was treated with nitrous acid by a modification of Bouveault's method (*Trans.*, 1898, lxxiii., 781), and chlorohydroxyisonicotinic acid obtained. This substance is identical with the product obtained by the action of soda on dichloroisonicotinic acid, as was shown by the melting point of the methyl ester. The acid, on treatment with aqueous ammonia, yielded a compound free from chlorine, which proved to be aminohydroxyisonicotinic acid, and by its interaction with phosphorus pentachloride the latter was converted into α-aminotetrachloropyridine, also identical with that mentioned above. These changes are thus shown:—



The reduction of dichloroisonicotinic acid to isonicotinic acid by hydrogen iodide has been described by Behrmann and Hofmann (*loc. cit.*, p. 2696). The authors show that an interesting intermediate compound, diiodoisonicotinic acid, is produced.

By heating the silver salt of *aa'*-dichloroisonicotinic acid in a stream of carbon dioxide, *aa*-dichloropyridine was obtained. This compound melts at 87—88°, and is identical with the dichloropyridine obtained by the interaction of pyridine and phosphorus pentachloride (*Trans.*, 1898, lxxiii., 433).

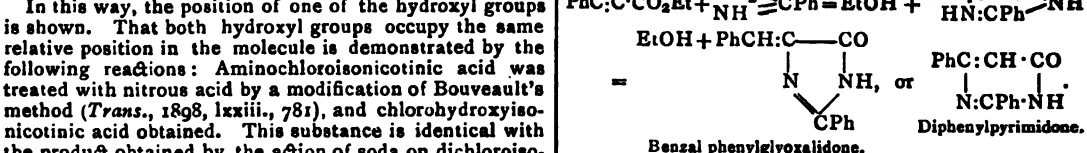
Discussion.

Dr. Ruhemann said that if the work of Behrmann and Hofmann left any doubt as to the position of the hydroxyl groups in citrazinic acid, it had been completely removed by the study of the action of ammonia on ethyl aconitate. The only explanation possible of this reaction was that ammonia effected the ring formation by condensing with the two carboxyl groups, and that the oxygen atoms in the citrazinamide thus were in the *aa'*-positions. Confirmatory evidence was given also by the formation, in an analogous manner, of the dicarboxylic acids derived from *aa'*-dihydroxypridine.

Mr. Sell, in reply, pointed out that, although by no means minimising the value of proofs of constitution derived from methods of formation, especially where the yield was approximately theoretical, he was of opinion that where (as in the cases cited by Dr. Ruhemann), something like 80 per cent of the materials are converted into substances as yet unidentified, it was most essential that independent and conclusive evidence should be brought forward.

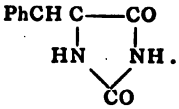
9. "*The Formation of Heterocyclic Compounds.*" By S. Ruhemann and H. E. Stapleton.

The authors have continued the investigation of the interaction of organic bases and ethereal salts of acids of the acetylene series, and have found that the action of bases on ethyl phenylpropiolate gives rise to heterocyclic compounds. In the case of benzamidine, when the reaction takes place at the temperature of the water-bath, besides the benzalphenylglyoxalidone previously described (Ruhemann and Cunnington, *Trans.*, 1899, lxxv., 954), *diphenylpyrimidone* is formed. This shows that benzamidine first reacts with the ethereal group of ethyl phenylpropiolate, forming a substituted amide, which subsequently condenses to a cyclic compound, thus:—

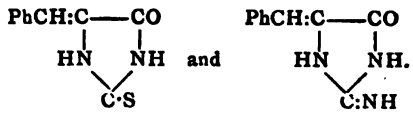


Benzal phenylglyoxalidone. Diphenylpyrimidone.

The compound formed with urea, previously described as a simple ureide, they now find to be benzalhydantoin,—



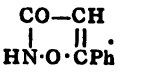
Similarly, thiourea and guanidine yield respectively,—



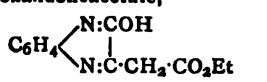
Benzal thiohydantoin. Benzal imidohydantoin.

The constitution of these substances is proved by their giving α-hydroxycinnamic acid when boiled with potash.

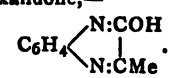
The authors further show that the compound previously described as phenylpropiolohydroxamic acid, is α-phenol-γ-isoxazolone—



With ethyl acetylenedicarboxylate, *o*-phenylenediamine gives ethyl quinoxalidoneacetate,—



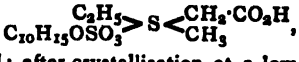
(m. p. 210°), which on hydrolysis loses carbon dioxide, giving methylquinoxalidone,—



The authors found that ethyl propenetetracarboxylate (formed by the union of the ethyl salts of malonic and acetylenedicarboxylic acids) reacts with ammonia, giving ethyl *aa'*-dihydroxypyridine-βγ-dicarboxylate, which, on hydrolysis with hydrochloric acid, loses the β-CO_2H group, and yields citrazinic acid.

10. "*The Space Configuration of Quadrivalent Sulphur Derivatives. Methylethylthetine Dextrocamphorsulphonate and Dextro-α-bromocamphorsulphonate.*" By William Jackson Pope and Stanley John Peachey.

On treating methylethylthetine bromide in aqueous solution with the corresponding quantity of silver dextrocamphorsulphonate, filtering and distilling off the water under reduced pressure, a crystalline residue of *methylethylthetine dextrocamphorsulphonate*,—



was obtained; after crystallisation at a low temperature from alcohol greatly diluted with ether, it forms colour-

less microscopic prisms melting at 115—117°. When pure, it is not hygroscopic, but is extremely soluble in water, alcohol, or acetone.

Methylethylthetine dextro-α-bromocamphorsulphonate,—

$$C_{10}H_{14}BrOSO_3 > S < \begin{matrix} C_2H_5 \\ CH_2\cdot CO_2H \\ CH_3 \end{matrix},$$

was prepared in a similar manner; it is sparingly soluble in cold acetone, and crystallises in silky white needles melting at 166—168°.

The molecular rotatory powers of these salts in dilute aqueous solution are the molecular rotatory powers of the optically active acid ions, so that the basic thetine group possesses no optical activity; no resolution of the asymmetric thetine into optically active components occurs, because more than one-half of the thetine bromide can be isolated as the pure optically active sulphonate. It is further most improbable that both salts are partially racemic and contain a dextro- and a lævo-thetine radicle, because partially racemic substances are of very rare occurrence.

The conclusion is drawn that, if sulphur really exists as a quadrivalent element in these salts, the four atoms directly and independently attached to the sulphur atom lie in the same plane as the latter, and that quadrivalent sulphur cannot lead to enantiomorphous configurations.

Previous attempts to prepare salts of asymmetric thetines and sulphonium compounds with optically active acids have invariably led to uncrystallisable products; the present are therefore the first observations from which information can be derived respecting the space configuration of quadrivalent sulphur compounds.

The investigation of these and allied substances is being continued.

*11. "*Nitrocamphane*." By M. O. Forster.

The compound, $C_{10}H_{16}O_2NBr$, obtained by the action of potassium hypobromite on camphoroxime (*Trans.*, 1899, lxxv., 1141), is a *bromonitrocamphane*,—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ CBr\cdot NO_2 \end{matrix},$$

although it gives Liebermann's reaction for nitroso-derivatives.

Nitrocamphane,—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ CH\cdot NO_2 \end{matrix},$$

prepared by reducing bromonitrocamphane with alcoholic potash, melts at 147—148°, and has the specific rotatory power $[\alpha]_D$ +4·5° in alcohol; it gives Liebermann's reaction for nitroso-derivatives, and is indifferent towards alcoholic ferric chloride.

Pseudonitrocamphane,—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ C:N(OH):O \end{matrix},$$

obtained on acidifying a solution of nitrocamphane in caustic potash, melts at 74°, and has $[\alpha]_D$ −95° (approximately); it develops a deep cherry-red colouration with ferric chloride, and changes spontaneously into the normal modification either in the solid or dissolved state. In the latter condition, this transformation is accelerated by the influence of piperidine, alkalis, and sunlight. It forms a *potassium* derivative, a *benzoyl* derivative, and a *pseudonitrole*.

Chloronitrocamphane,—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ CCl\cdot NO_2 \end{matrix},$$

produced when sodium hypochlorite acts on camphoroxime, or when chlorine water is added to a solution of nitrocamphane in caustic alkali, melts at 217°, and has $[\alpha]_D$ −53·1° in alcohol; it gives Liebermann's reaction for nitroso-derivatives.

Iodonitrocamphane,—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ CI\cdot NO_2 \end{matrix},$$

precipitated from solutions of nitrocamphane in potash by iodine dissolved in potassium iodide, melts at 179°, and has $[\alpha]_D$ −10·8° in alcohol; it gives Liebermann's reaction.

Hydroxylaminocamphane (*bornylhydroxylamine*,)—

$$C_8H_{14}\begin{matrix} CH_2 \\ | \\ CH\cdot NH\cdot OH \end{matrix},$$

prepared by reducing nitrocamphane with aluminium amalgam, crystallises from petroleum in small, oblong plates and melts at 154°; it dissolves in hot water, and the cold solution reduces ferric chloride, ammoniacal silver nitrate, and Fehling's solution, almost immediately.

12. "*The Absorption Spectra of Ammonia, &c.*" By W. N. Hartley, F.R.S., and James J. Dobbie, D.Sc., M.A.

It was shown by Soret, and subsequently by Hartley and Huntington, that the purest forms of commercial ammonia which could be purchased (even that stated to be "volcanic ammonia"), contained some substance which exhibited an absorption band which was believed to be due to traces of some constituent of gas-liquor. There was distinct evidence that the band was due to an impurity (*Phil. Trans.*, Part I., 1879, p. 267). An aqueous solution of ordinary ammonia, 150 m.m. in thickness, containing 2·5 grms. ammonia in 100 c.c., gives a broad absorption band between 1/λ 3694 (λ 2707) and 1/λ 4306 (λ 2322). This band becomes less marked when the ammonia is converted into ammonium chloride, and this recrystallised repeatedly. It is altogether absent from ammonia prepared from ammonium oxalate which has been carefully purified by crystallisation. It is also absent from ammonia prepared by the reduction of hydroxylamine hydrochloride. The authors conclude therefore that the absorption shown by ordinary aqueous ammonia is due to an impurity which is not of inorganic origin, but is more probably due to pyridine bases. They have, in fact, found that ordinary strong ammonia is contaminated with pyridine bases to the extent of about 0·00014 per cent.

They have measured also the continuous absorption of a normal solution of ammonia 200 m.m. and 100 m.m. in thickness. Methylamine hydrochloride and hydroxylamine hydrochloride show no selective absorption and are both highly diathinic. A solution of the former, 150 m.m. in thickness, containing 2·5 grms. methylamine in 100 c.c. distilled water, shows practically no absorption, whilst a layer of the latter of the same thickness and strength transmits the whole spectrum with the exception of a few lines at the extreme violet end.

Acetaldoxime and acetoxime show considerable general absorption, but no selective absorption. A solution of acetaldoxime 150 m.m. in thickness, containing 1·25 grms. acetaldoxime in 100 c.c. of water, absorbs all lines beyond 1/λ 3323 (λ 3009); a layer 25 m.m. in thickness of a solution containing 1 milligrm.-molecule dissolved in 20 c.c. of water gives a continuous spectrum to 1/λ 3952 (λ 2530). Acetoxime shows slightly greater absorption than acetaldoxime, due to the additional methyl group.

13. "*Isoamarine*." By Francis R. Japp, F.R.S., and James Moir, M.A., B.Sc.

In a recently published note (*Proc.*, 1899, xv., 211), the authors showed that both ordinary amarine and the isoamarine of Snape and Brooke (m. p. 198°) have the structure—

$$\begin{matrix} C_6H_5\cdot CH\cdot NH \\ | \\ C_6H_5\cdot CH-N \end{matrix} > C\cdot C_6H_5,$$

differing only in their configuration; the former being the meso-, the latter the racemic, form. The correctness of this view of the constitution of isoamarine was confirmed

by heating *r*-diphenylethylenediamine with benzoic acid, and subsequently by its re-solution into the enantiomorphs by Snape (*Proc.*, 1899, xv., 228).

Feist and Arnstein, however, by heating *s*-dibenzoyl-*r*-diphenylethylenediamine in a current of gaseous hydrogen chloride, claim to have prepared an isoamarine melting at 175°, structurally identical with the foregoing (*Ber.*, 1895, xxviii., 3177). As a third configuration—apart from those of the separate enantiomorphs, of which there can be no question here—is contrary to what theory predicts, we resolved to re-examine this compound.

We find that it melts at 198° instead of at 175°, and is in every respect identical with the isoamarine of Snape and Brooke.

The compound is so easy to purify that the wrong melting-point given by Feist and Arnstein is doubtless a mere inadvertence.

(To be continued).

PHYSICAL SOCIETY.

Annual General Meeting, February 9th, 1900.

Prof. Lodge, F.R.S., President, in the Chair.

The following Officers were elected to form the Council:—

President—Professor Lodge.

Vice-Presidents, who have filled the Office of President—Dr. Gladstone, Prof. Carey Foster, Prof. Adams, Lord Kelvin, Prof. Clifton, Prof. Reinold, Prof. Ayrton, Prof. Fitzgerald, Prof. Rucker, Sir W. Abney, Mr. Shelford Bidwell.

Vice-Presidents — Mr. Blakesley, Mr. Boys, Prof. Everett, Mr. Griffith.

Secretaries—Messrs. H. M. Elder and W. Watson.

Foreign Secretary—Prof. S. P. Thompson.

Librarian—Mr. W. Watson.

Treasurer—Prof. Callendar.

Other Members of Council—Prof. Armstrong, Dr. Atkinson, Mr. W. Baily, Prof. Glazebrook, Mr. E. H. Griffiths, Mr. S. Lupton, Prof. Perry, Mr. Swinburne, Prof. Threlfall, and Mr. J. Walker.

Mr. Addenbrooke asked if the Proceedings of the Society could be published with less delay.

The Chairman promised to try and have them printed sooner.

Prof. Lodge then delivered his Presidential Address on "*The Controversy Concerning Volta's Contact Force.*"

Those who take a metallic view of the Volta contact force are accustomed to deny that the Peltier evolution of heat measures the local E.M.F. existing at a junction; they assert that it measures the rate at which that same E.M.F. varies with temperature. In the thermodynamic equation connecting the Peltier effect with the variation of E. with temperature, the E. which varies is not necessarily that at the junction considered, but is the total E.M.F. of the circuit. The reversible heat at a specified junction is a measure of the metallic E.M.F. located there. Those who say it is a temperature variation of the E.M.F. beg the question by locating the whole E.M.F. of the circuit at the particular junction they are considering; usually an interface of zinc and copper. At a chemical junction the E.M.F. is not purely thermal, and hence is not measured by the Peltier effect; it is chiefly of chemical origin, and is calculable from the energy of combination of the materials on either side of the boundary. At a metallic junction there is no such chemical potentiality. A strong current may be passed across a zinc-copper junction for years and no brass is formed. It is therefore improbable that the chemical affinity of zinc for copper is the propelling influence which causes the E.M.F. located at such a junction. In showing the volta effect experimentally a trace of liquid can act detrimentally by forming a conducting bridge between the plates, across which the bulk of the electricity passes as the metals are being separated. The safest and clearest mode of expressing the Volta effect is that it consists in an opposite charge acquired by dry zinc and copper, while in metallic contact a charge which results from an E.M.F. of fixed value and is controlled solely by this E.M.F. and electrostatic capacity. It is undeniable that the order of the Volta force can be calculated from the differential heats of combination of the metals for oxygen, although it is doubtful whether it can be calculated from the heat of formation of brass. The opposing sides of the old controversy used to be called contact theorists and chemical theorists. Now the opposite sides are involved both in contact and both in chemical views. It is a question of which of several contacts is the effective one and what kind of chemical action or affinity is the active cause. Is it the contact and chemical affinity across the metal-metal junctions, or across the metal-air junctions? The opposite sides are thus metallic and dielectric. The metal-air force is of the order volts, the metal-metal force is of the order milli-volts.

When a piece of zinc is put in contact with a piece of copper the oxygen atoms which surround these bodies move slightly away from the copper and approach slightly nearer to the zinc. These slight motions produce the whole Volta effect. All that is necessary for the Volta effect is the inherent film on the surface, all the rest of the gas is mere dielectric, and might be substituted by a vacuum.

It was proposed by Prof. Perry and seconded by Prof. Armstrong that a meeting should be held to discuss the Address.

The meeting then adjourned until February 23rd.

NOTICES OF BOOKS.

The Early Metallurgy of Copper, Tin, and Iron in Europe; as Illustrated by Ancient Remains, and the Primitive Processes Surviving in Japan. By W. Gowland, F.S.A., F.C.S. London: J. B. Nichols and Sons. 1899. Pp. 56.

Although the primitive processes by which metals were extracted from their ores are generally dying out, they have a certain interest on account of their efficiency in spite of the rudeness of the appliances employed. The processes were, in principle, almost identical with those now in use; it is only the form of apparatus that has changed.

These ancient processes can be best studied in Japan, where, owing to the great conservatism and seclusion observed by that country until quite recently, they are (or were in 1872), still to be found in practical use. There are a number of quaint illustrations, showing the Japanese at work in their primitive ways of mining, baling water out of the mine, smelting, roasting, casting, &c., and comparing these methods with remains of ancient mining to be found in some parts of Great Britain, it is evident that those processes now in use in Japan are identical with those used in this country in prehistoric times.

The book makes interesting reading; one of the most striking thoughts to which it gives rise is, what remarkably cheap labour was to be obtained in those times.

Instruction in the Preparation of Chemical Compounds. ("Anleitung zur Darstellung Chemischer Präparate"). By Prof. Dr. H. Erdmann. Second edition, with 15 illustrations in the text. Frankfurt-a-M.: H. Bechhold. 1899. Pp. 92.

The object of this volume is to provide a text-book of practical instruction in the preparation of the inorganic compounds most frequently met with.

Thirty-five of the more common elements are dealt with, and details of the preparation of four or five com-

pounds of each are given. The tables at the end include a list of 75 elements with their atomic weights on two scales, viz., H=1, and O=16; as well as a register of authorities quoted, and a comprehensive index.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 4, January 22, 1900.

Metallic Borates.—L. Ouvrard.—Metallic borates are difficult to prepare. The author uses the following property for their preparation:—When boric anhydride acts on melted metallic chlorides, the chlorinated compounds are only formed to a very slight degree, still less in the case of the fluorides. The double fluorides of the metals with the alkalis are used as they are soluble and more easily fusible. Cadmium borate was one of the first the author prepared. Equimolecular parts of fluorhydrate of potassium fluoride and boric anhydride, to which was added a molecule of cadmium oxide, were placed in a platinum crucible. These were heated together for some time and then fused. Boron fluoride was evolved. When all the gas was given off and the whole was in tranquil fusion, it is allowed to cool slowly and taken up with water. Needles of tri-cadmic borate were left; these crystals are insoluble in water, but easily soluble in dilute acids. Analysis shows them to have the formula $B_2O_3.3CdO$.

New Method of Estimating Aluminium.—Alfred Stock.—When a mixture of iodide and iodate of potassium acts upon a solution of an aluminium salt, aluminium hydrate is precipitated and the corresponding quantity of iodine is liberated, according to the equation $Al(SO_4)_3+5KI+KIO_3+3H_2O = 2Al(OH)_3+3H_2SO_4+6I$. This is a reaction which begins very rapidly in the cold, but which is not complete for several days, especially with dilute solutions; the velocity of the reaction can, however, be accelerated if the iodine liberated during the reaction is removed by means of sodium thiosulphate, or by allowing the precipitation to take place when hot. By working on a boiling water-bath, the reaction is complete in a few minutes, and by removing the iodine at the same time by means of standard thiosulphate solution, a method of estimating aluminium can be devised—the precipitation being total, even in dilute solution.

MISCELLANEOUS

Formation of Hydrocyanic Acid by the Action of Carbonic Acid on Ferrocyanide of Potassium.—Guido Gigli.—If we pass a current of carbonic acid through a cold solution of ferrocyanide of potassium, there is no apparent effect; but if the solution is then submitted to distillation, it becomes cloudy as soon as the boiling point is reached, and we observe that the filtered liquid contains hydrocyanic acid. A fresh current of carbonic acid through the cloudy solution produces no effect, but if it is filtered the clear filtrate will then behave in the same manner as the original solution. This cycle of operations, action of carbonic acid gas, boiling, filtration, and cooling, can be repeated a large number of times; the quantity of ferrocyanide decomposed or of hydrocyanic acid produced in each operation is extremely small, but sufficient to interfere with the use of carbonic acid as a means for the detection of cyanide of potassium in the ferrocyanide (Fresenius's method). The greenish precipitate which is formed seems to be the product known under the name of "Everett's white salt."—*Chemiker Zeitung*, xxii., 775.

Preparation and Reduction of Dehydromucic Acid.—H. B. Hill.—The author easily obtained dehydromucic acid with a return of about 27 per cent, by heating saccharate of potash with four times its weight of hydrobromic acid ($d=1.49$), for ten hours in a flask fitted with a vertical condenser. The dehydromucic acid is then isolated by means of the calcium salt. The reduction of this acid furnishes two products, already described by M. Seelig, and which unite with bromine, giving the well crystallised compounds answering to the formulæ, $C_6H_6Br_2O_5,H_2O$ (fusible at 145°), and $C_6H_6Br_2O_5,2H_2O$ (fusible at 207°).—*Berichte der D. Ch. Ges.*, xxxii., p. 1221.

Hydracinnamoïn.—J. Thiele.—On reducing cinnamic aldehyde by means of zinc powder copper-plated with a solution of sulphate of copper, the author obtained hydracinnamoïn $C_6H_5.CH:CH.CH(OH)CH(OH)CH:CH.C_6H_5$, which crystallises in white needles, difficultly soluble in alcohol and ether, fusible at 153—154°. Hydrocinnamoïn reduces permanganate; in acetic solution it takes up bromine; heated with acids, or above its fusion point, it gives resins the same as when it is treated with an alcoholic solution of potash. It is with difficulty attacked by acetic anhydride. Dissolved in pyridine it furnishes, by the action of chloride of acetyl, a diacetate which crystallises in alcohol in small white prisms, fusible at 118—119°. Its dibenzoate, prepared in an analogous manner, occurs in fine colourless needles, fusible at 169—170°.—*Berichte der D. Ch. Ges.*, xxxii., p. 1296.

On the Peroxysulphate of Silver.—E. Mulder.—The electrolysis of sulphate of silver offers great difficulties on account of the very slight solubility of this salt, and further, it is necessary to operate in very dilute solutions (⅛ saturated), and to eliminate the SO_4H_2 formed, which tends to cause a reverse action. This last proviso is effected by continuous neutralisation by means of carbonate of silver, with the help of an automatic arrangement driven by clock-work. We thus obtain a microcrystalline compound, the peroxysulphate of silver, which is decomposed by warm water, with the precipitation of Ag_2O_2, disengagement of oxygen, and formation of sulphate of silver. This body is thus analogous to the peroxynitrate of silver, and can be represented by the formula $5Ag_2O_2.(SO_4.3O.Ag_2)$; it decomposes under the influence of sulphuric acid, and even spontaneously.—*Rev. d. Trav. Ch. P. B.*, xviii., p. 91.

An Ammonical Derivative of Fructose.—C. A. Lobry de Bruyn.—This derivative is obtained by dissolving 100 to 150 grms. of levulose in 100 c.c. of methylic alcohol saturated with ammonia, and leaving the solution to stand by itself, in contact with the air for several weeks. The product of the reaction is deposited in the form of amorphous grains, or crystalline leaves; the return is about 20 grms. for the above-mentioned quantities. It is purified by re-crystallising in water. The formation of this ammonical derivative takes place according to the equation, $C_6H_{12}O_6+NH_3=C_6H_9NO_4+2H_2O+H_2$; $\alpha_D = -75°$. This body is not basic, and its stability is much greater than that of the osamines; it does not give osazone, but reduces Fehling's solution warm, and is not attacked by nitrous acid. It is reduced by sodium amalgam. When treated with acetic anhydride and acetate of sodium, a tetracetate is obtained which crystallises in white flakes, fusible at 174°, $\alpha_D = -6.7°$, in chloroform solution at 2·4 per cent.—*Berichte*, xviii., 71.

Measurement of Solubilities at Various Temperatures.—B. Pawlewski.—The author proposes using a small piece of apparatus, formed of two vessels A and B, each fitted with a cork having two holes, and communicating one with the other by means of a syphon with a filtering plug, and with the outer air by two vertical tubes; the whole is plunged into a bath at a constant temperature. A is a test-tube containing the substance and its solvent; agitation is caused by blowing from B to A through one of the vertical tubes. When saturation is reached, we blow in the opposition direction; a certain

quantity of the saturated solution then passes into B, which is a conical flask. The whole apparatus is then withdrawn from the bath, and B is weighed. The vertical tubes are fitted with condensers, for cases in which the solvents are very volatile. The author gives as an example the determination of the solubility of chlorate of potassium in water. The following is an extract from the table:—

Temperature.	Weight of salt in 100 c.c. of saturated solution.	Weight of salt dissolved in 100 c.c. of water.	No. of parts of water to dissolve 1 part of salt.
0°	3·06	3·14	31·8
15°	5·11	5·35	18·5
50°	15·18	17·95	5·6
100°	35·83	55·54	1·8

—*Berichte*, xxxii., p. 1040.

Analysis of some Commercial Samples of Carbids of Calcium.—Henri Moissan.—Since the first publication of the preparation of crystallised carbide of calcium by means of the electric furnace, this substance has become of industrial importance, and a certain amount of difficulty is experienced in making it by the ton. A great deal of care is necessary in the preparation of the charge, and the behaviour of the large electrodes which have to carry such intense currents, &c. The disposal of, or utilisation of, the enormous amount of incandescent gases has also offered some difficulty. Carbide of calcium can now, however, be produced of a much better quality than formerly, but it is still of advantage to have it analysed. Theoretically, 1 grm. of carbide of calcium should give 349 litres of acetylene gas; but on decomposing a few samples with milk of lime, previously saturated with acetylene gas, the following volumes were obtained, reduced to 0° and 760 m.m.:—V = 292·80, 294·10, 301·30, 304·61, 307·72, 316·41, 318·77. If the carbide of calcium has not got a melted crystalline appearance the return of gas is much less, 228·60, 250·40, 260·30 for instance. The author has more particularly studied the insoluble residue left by the action of water on carbide of calcium. To render this more easy, the carbide was decomposed by sugar-water so as to keep the lime in solution as sucrate of lime. The residue was filtered and washed, then treated with alcohol, and finally with ether, and dried in vacuo at 40°. It consists principally of silicide of carbon, silicide of calcium and iron, and sometimes a little sulphide of calcium and graphite. Taken up with dilute HCl it loses a little weight, and iron, lime, alumina, and phosphorus is found in solution. The silicide of carbon and the graphite remain unattacked, and the sulphide of calcium has disappeared. By this treatment, and examining the residues under the microscope, the form in which most of the impurities exist was determined.—*Bull. de la Soc. Chim. de Paris*, Series 3, xxi., Nos. 20—21.

MEETINGS FOR THE WEEK.

TUESDAY, 20th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester.

WEDNESDAY, 21st.—Society of Arts, 8. "Artistic Copyright," by Edwin Bale.

— Microscopical, 8. Exhibition of Photo-micrographic and Projection Apparatus (with Lantern Illustrations), by J. W. Measures.

— Institution of Mining and Metallurgy, 8. "On the Development of Silver Smelting in Mexico," by Otto H. Hahn. "Segregation of Mine Accounts," by W. B. Middleton. "Notes on a Novel Association of Gold," by H. F. Collins.

THURSDAY, 22nd.—Royal Institution, 3. "Modern Astronomy," by Prof. H. H. Turner, M.A., F.R.S.

FRIDAY, 23rd.—Royal Institution, 9. "Recent Studies in Gravitation," by John H. Poynting, D.Sc., F.R.S.

— Physical, 5. Prof. R. W. Wood, of the University of Wisconsin, will Exhibit and Describe his:—(1) Photographs of Sound Waves and the Kinematographical Demonstration of the Evolutions of Reflected Wave-fronts; (2) A New Pseudoscope; (3) Diffraction Colour Photographs; (4) Artificial Parhelia.

SATURDAY, 24th.—Royal Institution, 3. "The Idea of Tragedy in Ancient and in Modern Drama," by W. L. Courtney, M.A., LL.D.

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MICHAELMAS TERM.—Monday, October 2, to Saturday, December 16.
LENT TERM.—Monday, January 8, to Saturday, Apr 7.
EASTER TERM.—Monday, April 30, to Saturday, July 28.
Forms of application can be had from the Assistant Secretary, Royal Institution, Albemarle Street, W.

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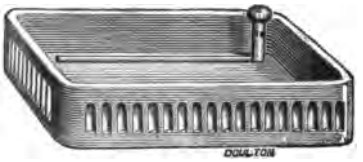

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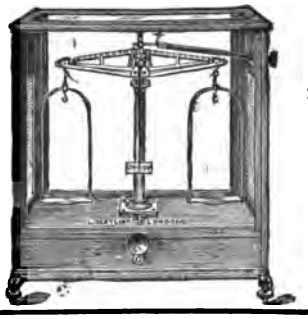

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VOL. LXXXI., No. 2100.

ON THE VISCOSITY OF ARGON AS AFFECTED BY TEMPERATURE.*

By Lord RAYLEIGH, F.R.S.

ACCORDING to the kinetic theory, as developed by Maxwell, the viscosity of a gas is independent of its density, whatever may be the character of the encounters taking place between the molecules. In the typical case of a gas subject to a uniform shearing motion, we may suppose that of the three component velocities v and w vanish, while u is a linear function of y, independent of x and z. If μ be the viscosity, the force transmitted tangentially across unit of area perpendicular to y is measured by $\mu du/dy$. This represents the relative momentum, parallel to x, which in unit of time crosses the area in one direction, the area being supposed to move with the velocity of the fluid at the place in question. We may suppose, for the sake of simplicity, and without real loss of generality, that u is zero at the plane. The momentum, which may now be reckoned absolutely, does not vanish, as in the case of a gas at rest throughout, because the molecules come from a greater or less distance, where (*e.g.*), the value of u is positive. The distance from which (upon the average) the molecules may be supposed to have come depends upon circumstances. If, for example, the molecules, retaining their number and velocity, interfere less with each other's motion, the distance in question will be increased. The same effect will be produced, without a change of quality, by a simple reduction in the number of molecules, *i.e.*, in the density of the gas, and is not difficult to recognise that the distance from which the molecules may be supposed to have come is *inversely as the density*. On this account the passage of tangential momentum *per molecule* is inversely as the density, and since the number of molecules crossing is directly as the density, the two effects compensate, and upon the whole the tangential force, and therefore the viscosity, remain unaltered by a change of density.

On the other hand, the manner in which this vicosity varies with temperature depends upon the nature of the encounters. If the molecules behave like Boscovich points, which exercise no force upon one another until the distance falls to a certain value, and which then repel one another infinitely (erroneously called the theory of elastic spheres), then, as Maxwell proved, the viscosity would be proportional to the square root of the absolute temperature. Or again, if the law of repulsion were as the inverse fifth power of the distance, viscosity would be as the absolute temperature.

In the more general case where the repulsive force varies as r^{-n}, the dependence of μ upon temperature may also be given. If v be the velocity of mean square proportional to the square root of the temperature, μ varies as—

$$\frac{n+3}{v^{n-1}},$$

a formula which includes the cases ($n=5$, $n=\infty$) already specified. If we assume the law already discussed—that μ is independent of density—this conclusion may be arrived at very simply by the method of "dimensions."

In order to see this we note that the only quantities (besides the density) on which μ can depend are m the mass of a particle, v the velocity of mean square, and k the repulsive force at unit distance. The dimensions of these quantities are as follows :—

$$\mu = (\text{mass})^1\,(\text{length})^{-1}\,(\text{time})^{-1},$$
$$m = (\text{mass})^1,$$
$$v = (\text{length})^1\,(\text{time})^{-1},$$
$$k = (\text{mass})^1\,(\text{length})^{n+1}\,(\text{time})^{-2}.$$

Thus, if we assume—

$$\mu \propto m^x \cdot v^y \cdot k^z \quad \ldots \quad (1),$$

we have—

$$1 = x+z, \quad -1 = y+(n+1)z, \quad -1 = -y-2z,$$

whence—

$$x = \frac{n+1}{n-1}, \quad y = \frac{n+3}{n-1}, \quad z = \frac{2}{n-1}.$$

Accordingly—

$$\mu = a \cdot m^{\frac{n+1}{n-1}} \cdot v^{\frac{n+3}{n-1}} \cdot k^{\frac{-2}{n-1}} \quad \ldots \quad (2),$$

where a is a purely numerical coefficient. For a given kind of molecule, m and k are constant. Thus—

$$\mu \propto v^{\frac{n+3}{n-1}} \propto \theta^{\frac{n+3}{2n-2}} \quad \ldots \quad (3).$$

The case of sudden impacts ($n=\infty$) gives, as already remarked, $\mu \propto v \propto \theta^{\frac{1}{2}}$. Hence k disappears, and the consideration of dimensions shows that $\mu \propto d^{-2}$, where d is the diameter of the particles.

The best experiments on air show that, so far as a formula of this kind can represent the facts, $\mu \propto \theta^{0\cdot77}$. It may be observed that $n=8$ corresponds to $\mu \propto \theta^{0\cdot78}$.

When we remember that the principal gases, such as oxygen, hydrogen, and nitrogen, are regarded as diatomic, we may be inclined to attribute the want of simplicity in the law connecting viscosity and temperature to the complication introduced by the want of symmetry in the molecules and consequent diversities of presentation in an encounter. It was with this idea that I thought it would be interesting to examine the influence of temperature upon the viscosity of argon, which in the matter of specific heat behaves as if composed of single atoms. From the fact that no appreciable part of the total energy is rotatory, we may infer that the forces called into play during our encounter are of a symmetrical character. It seemed therefore more likely that a simple relation between viscosity and temperature would obtain in the case of argon than in the case of the "diatomic" gases.

The best experimental arrangement for examining this question is probably that of Holman (*Phil. Mag.*, 1877, iii., p. 81), in which the same constant stream of gas passes in succession through two capillaries at different temperatures, the pressures being determined before the first and after the second passage, as well as between the two. But to a gas like argon, available in small quantities only, the application of this method is difficult. And it seemed unnecessary to insist upon the use of constant pressures, seeing that it was not proposed to investigate experimentally the dependence of transpiration upon pressure.

The theoretical formula for the volume of gas transpired, analogous to that first given by Stokes for an incompressible fluid, was developed by O. E. Meyer (*Pogg. Ann.*, 1866, cxxvii., p. 269). Although not quite rigorous, it probably suffices for the purpose in hand. If p_1, V_1 denote the pressure and volume of the gas as it enters the capillary, p_2, V_2 as it leaves the capillary, we have—

$$p_1V_1 = p_2V_2 = \frac{\pi t R^4}{16 \mu l}(p_1^2 - p_2^2) \quad \ldots \quad (4).$$

In this equation t denotes the time of transpiration, R the radius of the tube, l its length, and μ the viscosity measured in the usual way.

In order to understand the application of the formula for our present purpose it will be simplest to consider first the passage of equal volumes of different gases

* A Paper read before the Royal Society, January 18, 1900.

through the capillary, the initial pressures, and the constant temperature being the same. In an apparatus, such as that about to be described, the pressures change as the gas flows, but if the pressures are definite functions of the amount of gas which at any moment has passed the capillary, this variation does not interfere with the proportionality between t and μ. For example, if the viscosity be doubled, the flow takes place precisely as before, except that the scale of time is doubled. It will take twice as long as before to pass the same quantity of gas.

Although different gases have been employed in the present experiments, there has been no attempt to compare their viscosities, and indeed such a comparison would be difficult to carry out by this method. The question has been, how is the viscosity of a given gas affected by a change of temperature? In one set of experiments the capillary is at the temperature of the room; in a closely following set the capillary is bathed in saturated steam at a temperature that can be calculated from the height of the barometer.

If the temperature were changed throughout the whole apparatus from one absolute temperature θ to another absolute temperature θ', we could make immediate application of (4); the viscosities (μ, μ') at the two temperatures would be directly as the times of transpiration (t, t'). The matter is not quite so simple when, as in these experiments, the change of temperature takes place only in the capillary. A rise of temperature in the capillary now acts in two ways. Not only does it change the viscosity, but it increases the *volume* of gas which has to pass. The ratio of volumes is θ', θ; and thus—

$$\frac{\mu'}{\mu} = \frac{t'}{t} \times \frac{\theta}{\theta'} \quad \ldots\ldots\ldots\ldots \quad (5),$$

subject to a small correction for the effect of temperature upon the dimensions of the capillary. It is assumed that the temperature of the reservoirs is the same in both transpirations.

The apparatus is shown Fig. 1. The gas flows to and fro between the bulbs A and B, the flow from A to B only being timed. It is confined by mercury, which can pass through U connections of blown glass from A to C and from B to D. The bulbs B, C, D are supported upon their seats with a little plaster-of-paris. The capillary is nearly 5 feet (150 c.m.) in length, and is connected with the bulbs by gas tubing of moderate diameter, all joints being blown. E represents the jacket through which steam can be passed; its length exceeds that of the capillary by a few inches.

In order to charge the apparatus, the first step is the exhaustion. This is effected through the tap F, with the aid of a Toppler pump, and it is necessary to make a corresponding exhaustion in C and D, or the mercury would be drawn over. To this end the rubber terminal H is temporarily connected with G, while I leads to a common air-pump. When the exhaustion is complete, the gas to be tried is admitted gradually at F, the atmosphere being allowed again to exert its pressure in C and D. When the charge is sufficient, F is turned off, after which G remains open to the atmosphere, and H is connected to a manometer.

When a measurement is commenced, the first step is to read the temperatures of the bulbs and of the capillary; I is then connected to a force pump, and pressure is applied until so much of the gas is driven over that the mercury below A and in B assumes the positions shown in the diagram. I is then suddenly released so that the atmospheric pressure asserts itself in D, and the gas begins to flow back into B. The bulb J allows the flow a short time in which to establish itself before the time measurement begins as the mercury passes the connection passage K. When the mercury reaches L, the time measurement is closed.

One of the points to be kept in view in designing the apparatus is to secure long enough time of transpiration without unduly lowering the driving pressure. At the beginning of the measured transpiration the pressure in A was about 30 c.m. of mercury above atmosphere, and that in B about 2 c.m. below atmosphere. At the end the pressure in A was 20 c.m., and in B 3 c.m., both above atmosphere. Accordingly the driving pressure fell from 32 to 17 c.m.

Three, or, in the case of hydrogen, five, observations of the time were usually taken, and the agreement was such as to indicate that the mean would be correct to perhaps one-tenth of a second. The time for air at the temperature of the room was about ninety seconds, and for hydrogen forty-four seconds, but these numbers are not strictly comparable.

When the low temperature observations were finished, the gas was lighted under a small boiler placed upon a shelf above the apparatus, and steam was passed through the jacket. It was necessary to see that there was enough heat to maintain a steady issue of steam, yet not so much as to risk a sensible back pressure in the jacket. The time of transpiration for air was now about 139 seconds. Care was always taken to maintain the temperature of the bulbs at the same point as in the first observations.

There are one or two matters as to which an apparatus on these lines is necessarily somewhat imperfect. In the high temperature measurements the whole of the gas in the capillary is assumed to be at the temperature of boiling water, and all that is not in the capillary to be at the temperature of the room, assumptions not strictly compatible. The compromise adopted was to enclose in the jacket the whole of the capillary and about 2 inches at each end of the approaches, and seems sufficient to exclude sensible error when we remember the rapidity with which heat is conducted in small spaces. A second weak point is the assumption that the instantaneous pressures are represented by the heights of the moving mercury columns. If the connecting U-tubes are too narrow, the resistance to the flow of mercury enters into the question in much the same way as the flow of gas in the capillary. In order to obtain a check upon this source of error the apparatus has been varied. In an earlier form the connecting U-tubes were comparatively narrow; but the result for the ratio of viscosities of hot and cold air was substantially the same as that subsequently obtained with the improved apparatus, in which these tubes were much widened. Even if there be a sensible residual error arising from this cause, it can hardly affect the comparison of temperature-coefficients of gases whose viscosity is nearly the same.

I will now give an example in detail from the observations of December 21 with purified argon. The times of transpiration at the temperature of the room (15° C.) were in seconds—

104⅜, 104½, 104⅜. Mean, 104·67.

When the capillaries were bathed in steam, the corresponding times were—

167½, 167½, 167⅝. Mean, 167·58.

The barometer reading (corrected) being 767·4 m.m., we deduce as the temperature of the jacket 100·27° C. Thus $\theta = 287{\cdot}5$, $\theta' = 372{\cdot}8$. The reduction was effected by assuming—

$$\frac{t'}{t} = \left(\frac{\theta'}{\theta}\right)^{x} \quad \ldots\ldots\ldots\ldots \quad (6).$$

With the above values we get—

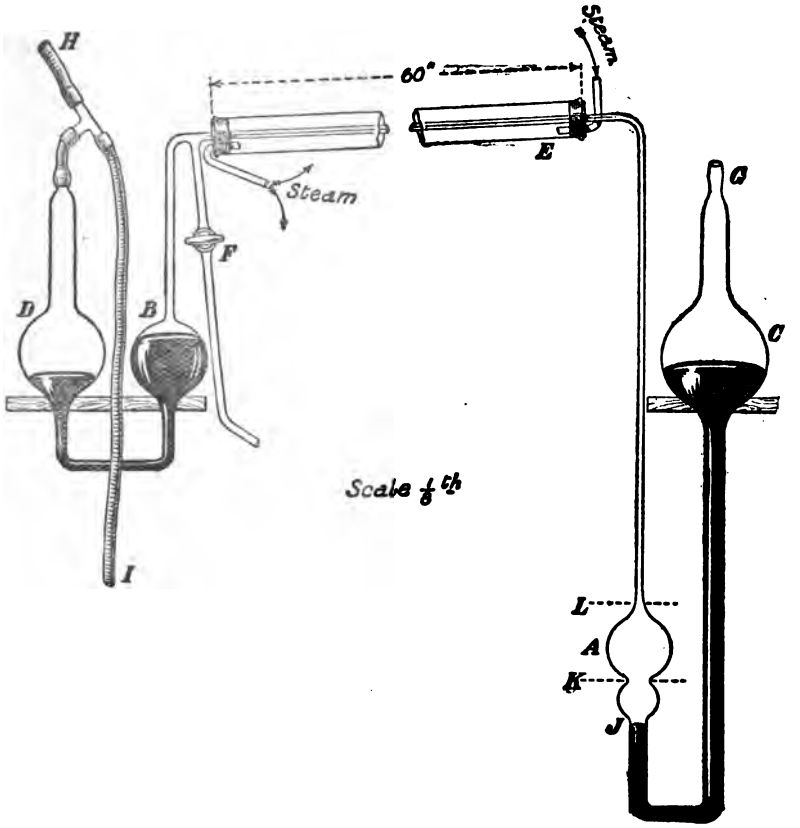
$$x = 1{\cdot}812.$$

As appears from (5), the integral part of x relates merely to the expansion of the gas by temperature. If we take—

$$\frac{\mu'}{\mu} = \left(\frac{\theta'}{\theta}\right)^{n} \quad \ldots\ldots\ldots\ldots \quad (7),$$

we get—

$$n = 0{\cdot}812.$$



This number is, however, subject to a small correction for the expansion of the glass of the capillary. As appears from (4), the ratio μ', μ as used above requires to be altered in the same ratio as that in which the glass expands by volume. The value of n must accordingly be increased by 0·010, making—

$$n = 0{\cdot}822.$$

The following table embodies the results obtained in a somewhat extended series of observations. The numbers given are the values of n in (7), corrected for the expansion of the glass:—

Air (dry)	0·754
Oxygen	0·782
Hydrogen	0·681
Argon (impure)	0·801
Argon (best)	0·815

In the last trials, the argon was probably within 1 or 2 per cent of absolute purity. The nitrogen lines could no longer be seen, and scarcely any further attraction could be effected on sparking with oxygen or hydrogen.

It will be seen that the temperature change of viscosity in argon does not differ very greatly from the corresponding change in air and oxygen. At any rate the simpler conditions under which we may suppose the collisions to occur, do not lead to values of n such as 0·5, or 1·0, discussed by theoretical writers.

I may recall that on a former occasion (*Roy. Soc. Proc.*, January, 1896) I found the viscosity of argon to be 1·21 relatively to that of air, both being observed at the temperature of the room.

Chemical Society.—An extra Meeting of the Society will be held on Thursday, March 8th, when a Lecture "On Recent Researches on Nitrification" will be given by Professor Warington, F.R.S. The Chair will be taken at 8.30 p.m.

THE PREPARATION AND PROPERTIES OF CRYSTALLISED PHOSPHIDE OF CALCIUM.

By HENRI MOISSAN.

By allowing the vapour of phosphorus to react on lime heated to redness, Paul Thenard obtained an amorphous product of a cinnabar-red colour, the composition of which answered to the formula P_2CaO (P. Thenard, *Ann. Chim. Phys.*, 1895, xiv., 12). This new body, which he called phosphide of lime, decomposed in the presence of water, and the reaction enabled him to establish the existence and the properties of the different hydrides of phosphorus. Previously to this, Dulong had described a general method for the preparation of phosphides by the action of the vapour of phosphorus on the metal. These experiments were repeated by Vigier.

This latter process was only applicable to the preparation of phosphide of calcium on the condition of the metal being pure, which had not been achieved before our researches on the subject.

Preparation of Crystallised Phosphide of Calcium by the Electric Furnace.

Phosphide of calcium can be prepared in the electric furnace, by the reduction of tricalcic phosphate by means of carbon. This operation, however, requires certain precautions, on account of the relatively easy decomposition of phosphide of calcium at the high temperature of the electric furnace.

We first prepared pure tricalcic phosphate by precipitation. This compound, after desiccation, was calcined in a Perrot furnace, then reduced to a fine powder, and finally mixed with carbon in the following proportions:—

Tricalcic phosphate	310 parts
Lamp-black	96 „

A small quantity of essence of turpentine was added to this mixture, so that it could be moulded into small cylinders, under pressure. These were then calcined in the Perrot furnace in a crucible brasqued with lamp-black.

The intimate mixture of phosphate of calcium and carbon is then placed in the crucible of the electric furnace and submitted to the action of an arc of 590 ampères and 45 volts, for four minutes.

On cooling, the melted mass is withdrawn from the crucible; it should not adhere to the sides, and will only do so when the heating has been carried on too long. In such a case the mass of phosphide of calcium will be contaminated round the edges with a small quantity of carbide of calcium. If, on the other hand, the heating has been insufficient, the phosphide will be mixed with melted unreduced phosphate, in the midst of which can be plainly distinguished the red crystals of the phosphide.

During this operation in the electric furnace only a very small quantity of phosphorus vapours is given off. But if we continue heating after the reduction is complete, the phosphide of calcium formed is in its turn decomposed, the phosphorus is distilled in abundance, and its vapour burns on escaping from the furnace. Finally, the calcium unites with the carbon, and there only remains carbide of calcium, which contains but a very small quantity of phosphorus.

Thus, as we remarked above, when we wish to obtain pure phosphide of calcium by this method, it is important not to continue the heating for too long.

We can also obtain fused phosphide of calcium, by heating, in a carbon crucible in the electric furnace, P. Thenard's phosphide of lime, prepared by the action of a large excess of phosphorus vapour on lime kept at a red heat. With an arc of 800 ampères and 50 volts, fusion takes place in seven or eight minutes. A well-melted, deep red mass remains, contaminated with carbide of calcium.

Preparation of Amorphous Phosphide of Calcium, by the Direct Union of Phosphorus and Calcium.

To effect this synthesis, a Bohemian glass tube is taken, closed at one end, and at the bottom there is placed a fair quantity of well-dried red phosphorus. A porcelain boat, placed in the lower part of the tube, contains calcium in the form of small crystals. The air is exhausted from the tube, which is then heated to a dull red, where the calcium is, and over which the fumes of phosphorus soon spread. Incandescence is produced, and very soon the whole of the metal is transformed into phosphide.

This last compound has the same formula as that obtained by means of the electric furnace, as we shall show later on.

Properties.

Phosphide of calcium, whether prepared in the electric furnace or by the direct union of the two elements, has the same colour when examined under the microscope in a state of fine division. It occurs in reddish brown fragments, of which the colour approaches that of nitride of calcium. When it has been prepared in the electric furnace it has a crystalline fracture, and, further, when the reduction has not been complete, we meet with distinctly formed crystals of a deep red colour, in the middle of the melted phosphide.

Phosphide of calcium is not easy to fuse; we have only succeeded in melting it by means of the electric furnace. In the combination of phosphorus with calcium, a compound the formation of which always takes place with incandescence, we only obtain an amorphous mass.

At the temperature of softening Bohemian glass, phosphide of calcium dissociates slowly *in vacuo*. The escape of a small quantity of phosphorus vapour is very marked. The tension of dissociation only reaches a few millimetres; its density at +15° is 2·51.

At 900° phosphide of calcium is not sensibly changed in an atmosphere of hydrogen.

Chlorine does not react on phosphide of calcium at all in the cold, but it suffices to slightly raise the temperature, to about 100° for example, to start the reaction, which then proceeds with lively incandescence.

Chloride of calcium is formed, as well as fumes of chloride of phosphorus. The action of bromine is in every way comparable with the action of chlorine, and the vapour of iodine attacks this phosphide at about a dull red heat.

The combustion of phosphide of calcium in oxygen takes place towards 300° with lively incandescence, forming lime and phosphoric anhydride. Sulphur reacts in the same manner at about 300°; the decomposition of the phosphide takes place with a considerable disengagement of heat, and the production of a yellow sublimate and formation of sulphide of calcium.

The action of nitrogen on phosphide of calcium seemed to us to be particularly interesting. In fact, if the nitrogen was able to drive the phosphorus out of this compound, we could easily pass from the phosphide to the nitride of calcium.

We have already shown that nitride of calcium is easily decomposed on contact with water, giving ammonia, a reaction susceptible of commercial application.

But the heat of formation of the phosphide of calcium appears to be higher than that of the nitride, as all our experiments up to a temperature of 900° have been fruitless. Phosphide of calcium, heated in an atmosphere of nitrogen to 900°, does not vary in weight, and does not give off any ammonia on its decomposition by water.

At 1200° nitrogen partially combines with calcium, with the separation of a small quantity of phosphorus; but the reaction is far from being complete, and the residue in the presence of water gives a mixture of ammonia and phosphoretted hydrogen, the latter being in great excess. We are here in the presence of a dissociation of phosphide of calcium.

When heated in the vapour of arsenic, at the temperature of melting glass, phosphide of calcium was not attacked. Boron and carbon are without action at a temperature of 700°. In the electric furnace it is no longer the same, and the carbon displaces the phosphorus from phosphide of calcium, producing melted carbide of calcium.

The gaseous hydracids react with energy on phosphide of calcium. Hydrochloric acid attacks it with incandescence, at a red heat. Towards 700° sulphuretted hydrogen does not attack it to any noticeable extent. It is the same with ammonia gas. The reaction of water with this body is very important. On contact with cold water phosphide of calcium is at once decomposed, with the formation of hydrate of lime and phosphoretted hydrogen gas. If the phosphide of calcium is in crystalline fragments, the hydrate of lime produced hinders the reaction; if, on the contrary, the phosphide is in powder, the reaction is violent.

When the phosphide has been sufficiently heated in the electric furnace, the phosphoretted hydrogen produced in the decomposition by water is not spontaneously inflammable on contact with the air. This new phosphide is thus very different from the body produced by P. Thenard. However, the reaction of the decomposition is still complex, and we do not obtain the whole of the phosphorus from the phosphide as phosphoretted hydrogen, PhH_3. Some samples, which were less heated, gave with water a phosphoretted hydrogen contaminated with free hydrogen. When, on the contrary, the phosphide had been carried to a very high temperature, hydrogen was no longer given off, but a small quantity of acetylene gas was collected. We shall give all the figures of these analyses in the paper we are about to publish in the *Annales de Chimie et de Physique*.

Acids will not attack this phosphide, except in respect of the water they may contain; thus, with monohydrated nitric acid, the attack is almost *nil* in the cold; when heated it is very slow. With ordinary nitric acid the

oxidation is rapid in the cold—nitrous fumes are produced as well as a spontaneously inflammable gas.

In the same way, fuming sulphuric acid does not attack phosphorus in the cold, while the decomposition is violent with dilute sulphuric acid.

Absolute alcohol, ether, benzene, and essence of turpentine do not react at the ordinary temperature.

On the other hand, oxidising agents attack with violence, both the chlorate and bichromate of potassium in fusion oxidise it with incandescence. If we gently heat a mixture of permanganate of potassium and powdered phosphide of calcium, the reaction is violent, and produces incandescence and explosion. Both the protoxide and binoxide of nitrogen oxidise it in the same manner when warmed, with lively incandescence. Lime and a very small quantity of nitride are formed.

Analysis of Phosphide of Calcium.

Phosphide of calcium was decomposed by direct oxidation by means of fuming nitric acid. The metal was precipitated in the state of sulphate of calcium in alcohol, and the phosphoric acid was then weighed in the form of ammonio-magnesium phosphate. In this manner we found the following figures:—Ca, 65·82, 65·71, 65·38, and 65·40; Ph, 33·79, 33·92, and 33·85,—theory for Ph_2Ca_3 = Ca, 85·93; Ph, 34·06.

These figures must be compared with those given by synthesis. The apparatus was arranged as before; the boat was weighed empty, then filled with calcium, and finally when containing the phosphide of calcium formed; the figures were:—Ca, 66·31 and 66·12; Ph, 33·53 and 33·41.

Conclusions.

To sum up, the reduction of the tricalcic phosphate by carbon gives, under certain conditions, a crystallised phosphide of calcium, of a deep red colour, answering to the formula Ph_2Ca_2. The most curious reaction of this new compound is its easy decomposition of cold water with the formation of slaked lime and phosphoretted hydrogen.

It is of sufficient interest to justify the remark that a large number of binary compounds of calcium possess this curious property of decomposing cold water and giving a hydrated oxide, and a gaseous compound of hydrogen united to the other element of the binary compound.

Hydride of calcium decomposes cold water with the evolution of hydrogen. Carbide of calcium decomposes cold water with the evolution of acetylene. Nitride of calcium gives the same reaction with the production of ammonia. We now see that phosphide of calcium can also give phosphoretted hydrogen. Finally, M. Lebeau has only just announced that the crystallised arsenides and antimonides of calcium behave in the same manner, giving the corresponding hydrides or the products of their decomposition, if the hydrides are unstable at the temperature of the experiment. The generality of this reaction appears to be very interesting.

We would finally remark that most of these new compounds have been prepared by means of the electric furnace.—*Bull. Soc. Chim.*, Series 3, vol. xxi., No. 22.

Action of Alkalis on Sugars.—C. Lobry de Bruyn and W. A. van Ekenstein.—Maltose heated to 100° with normal KOH for three hours, gives first glucose, which in its turn is transformed into mannose. The solution further contains a non-fermentable product which hydrolysed with a dilute acid forms glucose. Melibiose, like lactose, gives, under the same conditions, galactose; but no writers have been able to put forward any evidence as to the formation of glucose. There appears to be formed, as in the case of maltose, an anhydride of glucose which by hydrolysis regenerates the former.—*Revue Trav. Chem. P.-B.*, xviii., p. 147.

ON THE HEAT OF FORMATION OF QUICKLIME FROM ITS ELEMENTS.

By HENRI MOISSAN.

THE heat of formation of quicklime from its elements, calcium and oxygen, has been determined by Thomson (*Thermochemische Untersuchungen*, vol. iii.), who found it to be +131·5 cal. But the results given by him offer several points of uncertainty, by reason of the impurities in his calcium, and also on account of the method used.

These experiments were made with calcium containing iron, attacked in the calorimeter by a solution of hydrochloric acid, and the weight of the calcium was deduced from the volume of hydrogen given off. Further, the operation was carried out in the presence of petroleum, which necessarily complicated the experiment.

We came to the conclusion that it was indispensable to repeat this determination with the pure metal which was prepared by the help of its solubility in sodium. For these experiments, we used the calorimeter and the methods described by M. Berthelot.

When calcium, obtained in small crystals, is thrown on to the surface of water, it decomposes this latter very rapidly, and if the quantity of water is not very great, or if the calcium is in the form of fine powder, incandescent phenomena may take place. Further, each particle of calcium becomes surrounded with a cushion of hydrogen, and runs about on the surface of the liquid. It is therefore impossible, under the circumstances, to make any thermometric determination. On the other hand, if the metal is melted, or agglomerated by pressure, the decomposition on contact with water will be much slower, on account of the slaked lime which quickly encases the metal and retards the reaction.

A cylinder of calcium weighing about 1 grm. sometimes requires more than an hour's contact with water to become transformed into slaked lime, and the experiment is much too long to be applied to a calorimetric measurement.

To prevent these drawbacks we endeavoured to make only slightly agglomerated—that is to say, porous—cylinders, the transformation of which in the presence of water would not take more than three or four minutes.

But in these new experiments, as soon as the height of the cylinder was more than one-third of its diameter, the compression was no longer uniform, and the metal became disaggregated during the attack by water; small particles became detached and ran about the surface of the liquid.

Finally, the preparation of the cylinder always necessitated a more or less prolonged handling of the calcium in the presence of air, which may give rise to a superficial oxidation with the formation of a small quantity of slaked lime.

We finally adopted the following method of procedure:—

Pure calcium in small crystals is placed in a cylindrical platinum tube, 0·04 metre long and 0·01 metre in diameter, formed of two parts fitting tightly one in the other. This tube is pierced with a large number of small holes of about 0·25 m.m. in diameter. When this platinum cylinder is thrown into water a very small quantity of liquid penetrates the interior, and the disengagement of hydrogen which takes place immediately prevents the admission of any more water. For this reason the decomposition is never too violent, and the reaction can be regulated with ease.

In the preliminary experiments we found that 1 grm. of calcium was completely attacked in three or four minutes. At the end of this time not a trace of unoxidised metal remained inside the platinum cylinder. At the end of the experiment the water became milky, and contained slaked lime in suspension, which indicated a complete saturation of this base.

To get the temperature of the calcium in equilibrium with that of the water in the calorimeter, the platinum

tube was placed in a tube of very thin glass of slightly greater volume, sealed before the blowpipe, and kept for a whole night at the bottom of the calorimeter filled with water. The next morning the glass tube was broken at the moment of commencing the experiment, and the variations of temperature then noted.

The weight of the calcium used was determined in two different manners:—

1. By weighing it in its natural state.

2. By estimation in the state of lime at the termination of the experiment. These two determinations give very close results.

It should be added that all the manipulation of the calcium, for filling the cylinder, should be performed at the bottom of a large glass vessel filled with dry carbonic acid gas.

In this manner we obtained for the reaction, $Ca+2H_2O=H_2+CaO_2H_2$, saturated solution, in two successive experiments, 94·74 cal. and 93·44 cal.; which gives a mean of 94·10 cal.

From this it is easy to calculate the heat of formation of lime from its elements by means of the two following cycles, in which the heat disengaged is the same:—

$Ca+O=CaO$ anhydrous..	x cals.
$CaO+Aq=CaO_2H_2$ sat. sol... ..	+18·1 (B) ,,
$H_2O=H_2+O$	−69·0 (B) ,,
$Ca+2H_2O=H_2+CaO_2H_2$ sat. sol.	94·10 ,,

from which we get—

$$x+18·1-69=94·10 \text{ cal.}$$
$$x=145·0 \text{ ,,}$$

a figure which is decidedly higher than that obtained by Thomson. From this figure we can immediately calculate those relating to the calcic hydrate:—

$Ca+O_2+H_2=CaO_2H_2$ solid..	+229·1 cal.
$Ca+O_2+H_2=CaO_2H_2$ sat. sol.	+232·1 ,,

We must explain that, according to these experiments, the heat of formation of the lime is greater than that of the oxides of potassium (+98·2), and of sodium (+100·9), which establishes the fact that calcium ought to displace sodium and potassium from their oxides; this actually occurs, as we have already shown when dealing with the chemical properties of calcium (*Comptes Rendus*, cxxvii., p. 584)

If we look over the heats of formation of the different oxides (Berthelot, *Thermochimie*, i., p. 710), we see that lithia, Li_2O, gives off +141·2 cal., in its production from its elements; although this figure is very close to that of quicklime, still it is lower.

We therefore thought it would be of interest to attempt to replace the lithium in lithia by metallic calcium. We did not, however, forget that we ought not, without certain reservations, to apply the results obtained at the ordinary temperature to reactions produced at a red heat in the dry way.

The experiment was performed in the following manner: A mixture of anhydrous lithia and crystals of calcium was placed in a tube of combustion glass. The air was exhausted by means of a Sprengel pump, and the mixture heated; the reaction took place at a dull red heat without incandescence, and the brilliant lithium ring was seen to condense on the cool part of the glass tube. Thus, as foreseen by theory, calcium displaces lithium from its anhydrous oxide.

If we consider the heats of formation of other oxides, we notice that $Mg+O=143·4$ cal.; it would here again appear that calcium ought to displace the magnesium. Now, this is not the case; we heated a mixture of quicklime and powdered magnesium in an iron crucible with a screw stopper, in the presence of an excess of sodium. This latter metal has the property, as we have already shown, of dissolving calcium when heated.

After having heated the mixture to redness for a few instants, the cake of sodium, which is found in the upper part of the crucible after cooling, contained crystals of calcium easily separated by means of anhydrous alcohol. Winkler's experiments have already made this known (*Berichte*, 1890, p. 122).

To sum up, magnesium easily displaces calcium from quicklime at a red heat.

We thought that the interpretation of this experiment might lead to the opinion that the heat of formation of anhydrous magnesia, determined by Thomson (*Thermochemische Untersuchungen*, iii., p. 241), was a little low.

At the period when M. Thomson made these determinations, metallic magnesium was not prepared in a great state of purity.

There would therefore be nothing surprising if this experiment had given a calorific value some units lower than the true figure.

We shall continue the study of this determination.—*Bull. Soc. Chim.*, Series 3, xxi., No. 22.

ON NICKEL-BRONZE ALLOYS.

By SERGIUS KERN, M.E., St. Petersburg.

LAST spring and summer we made experiments, which we are still continuing, on some nickel-bronze alloys; our aim was to produce castings from such alloys, to be used in mechanical work, especially for different fittings in high-pressure marine boilers.

The alloys rust very slightly, and in several cases are to be preferred to steel castings, more especially as the nickel-bronze castings made from our alloys No. I. and No. II. give very good results when mechanically tested.

Alloy No. I. corresponds to the English Admiralty's rules prescribed for ship-building steel, and alloy No. II. corresponds to the rules for steel castings (tensile strength 28 to 35 tons per square inch, with an elongation of 15 per cent in 2 inches).

Alloy No. I.

Copper	70·0 per cent.
Nickel	17·5 ,,
Zinc	12·5 ,,
	100·0

This alloy in the cast state gives a tensile strength of 26 tons, and 23 per cent of elongation, in 2 inches. Fracture, fibrous. Bending test, 67° over a radius of 1¼ inch.

Alloy No. II.

Copper	70·0 per cent.
Nickel	20·0 ,,
Zinc	10·0 ,,
	100·0

This alloy gives excellent results: tensile strength 36 tons, with an elongation of 14 to 17 per cent, in a length of 2 inches. Fracture, also fibrous. Bending test, 35—40° over a radius of 1⅛ inch.

Drop Tests.

Weight of monkey, 18 kilogrms. Specimens from alloys Nos. I. and II., with a section of 30 m.m. square, gave a bend to a right angle, without fracture, after eleven consecutive drops of the monkey. The height of the fall of the monkey at the first drop was 1 metre and 1·5 metre at the eleventh drop.

We prepare our alloys Nos. I. and II. in the same manner, using ordinary copper crucible furnaces; each furnace contains one crucible, with a charge of 150 pounds. Half of the copper is melted beforehand with all the zinc; the alloy is poured into thin plates, ½ of an inch thick, which are next sheared into small pieces.

For preparing the alloys we proceed as follows:—On the bottom of the crucible we place some nickel (in the form of cubes), next some copper-zinc alloy, and on the

latter some copper, going on in such a way till the crucible is filled up. On the top of the charge a layer of charcoal is placed. The melting of the 150 pounds charge takes about five hours.

The alloy is run into flat open moulds, and the slabs obtained are re-melted twice before being used for castings. At each melt about 0·75 per cent of zinc is added to the melted alloy, when the crucible is out of the furnace and ready to be poured into the mould.

The moulding is done exactly in the same way, concerning the sand, as when casting copper articles, but, as the alloys have a notable shrinkage in setting down, top ends must be placed on the castings, as is done with steel castings. After pouring the metal, the open top ends must be immediately covered with coarsely ground charcoal.

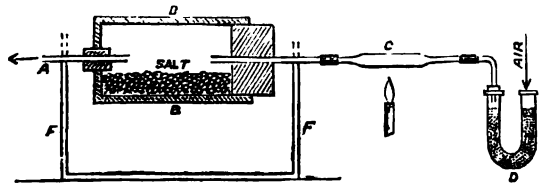
February, 1900.

A NEW DEVICE FOR DRYING CRYSTALLISED SALTS.

By F. H. GETMAN, F.C.S.

Being at a loss for a clay tile some days since, when preparing some antimony sulphate, I was forced to devise some form of "dryer" from the materials at hand. My first apparatus was so satisfactory that I was led to improve upon it, and now I feel that it may be of service in other laboratories.

The apparatus is shown in the accompanying sketch, where B B represents a large-sized porous cup through the bottom of which is bored a central hole large enough to receive a good-sized single-hole cork. The mouth of the cup is also fitted with a cork through which passes a single hole, the axis of which passes through the hole in the bottom. These corks are fitted with glass tubing, as shown, and the whole placed in a frame, F F, which may be made from an ordinary box. The tube passing



through the large cork is connected either directly with the drying tube, D, or with D through the hard glass tube C. The tube A is connected with an aspirator. The salt to be dried is placed in B B, the corks inserted tight, the cup placed in its supports, connections made with the drying tube, and then the aspirator started at A.

From time to time the cup is slowly turned, thus exposing both a fresh surface of cup to the salt, and also a fresh surface of salt to the current of air. If it is desired to use warm air, the tube C may be inserted in the train and heated to any desired temperature. This arrangement will be found vastly superior to the clay tile, and, owing to the simplicity of construction, can easily be made in any laboratory however small.

Chemical Laboratory, Stamford High School,
Stamford, Conn., U S.A.,
January 30, 1900.

Royal Institution.—On Thursday next (March 1st) at Three o'clock, Dr. Charles Waldstein, Slade Professor of Fine Arts, University of Cambridge, will deliver the first of a course of three Lectures at the Royal Institution, on "Recent Excavations at the Argive Horæum in Greece;" and on Saturday (March 3rd) Lord Rayleigh, Professor of Natural Philosophy, R.I., will begin a course of six Lectures on "Polarised Light."

NOTE ON THE

DIRECT COMBUSTION OF METALLIC ALLOYS.

By HARRY BREARLEY.

The estimation of carbon by igniting the metal in a stream of oxygen is said to have originated with Berzelius or Mulder. The difficulties of the process are practical ones; the finely-divided sample or the extremely high temperature required generally occasioning more trouble than a preliminary separation of the carbon. Where the sample is already in powder, however, or must be powdered for other operations, one objection to direct combustion no longer holds, and in such cases it seems worth while to enquire with what success the direct combustion may be resorted to when only easily available temperatures are used.

The term "direct combustion" is applied generally to the estimation of carbon in metals without previously separating it. There are processes of this kind, both wet and dry, in which a large variety of reagents are used; it is therefore necessary to say that here "direct combustion" refers to the simple ignition of the sample in a stream of oxygen without any reagent whatever.

Each of the following samples were passed through a sieve having 90 meshes to the linear inch, and, so as to indicate with what alloys the process is serviceable, the ignitions were limited to one hour, even when CO_2 was being *slowly* evolved at the end of that time. The furnace was of the ordinary Bunsen type, and the porcelain tube was supported in asbestos arches (Chemical News, lxxvii., 5). A temperature was thus obtained which made it impossible or very difficult to at once distinguish the boat on looking into the tube. This is hotter than combustion tubes generally are, but by no means hotter than can readily be attained with any other type of furnace, using the asbestos arches (obtainable from Becker, Hatton Wall, London, E.C.).

Material.	Direct ignition.	Actual C percentage.
Tungsten (metallic)	2·47	2·47
" "	0·65	0 64
" "	3·41	3·46
Ferro-tungsten (85 per cent) ..	5·75	5·64
" (50 ") ..	1·09	1·09
Ferro-silicon	2·04	2·00
"	1·59	1·55
12 per cent silicon-spiegel	2·31	2·35
12 " "	2·30	2·26
20 " spiegel	6·01	6·11
20 " "	4·94	5·02
Ferro-silico-molybdenum	1 58	1·56
Molybdenum (metallic)	4·69	4·72
70 per cent molybdenum-nickel ..	2·94	2·90
70 " tungsten-nickel..	1·00	1·32
80 " ferro-manganese . ..	2·82	7·01
60 " ferro-chromium	1·04	8·69
70 " chrome-nickel	1·53	6·65
Chromium (metallic)	0·44	5·13

Of those alloys set forth in the first part of the table, the 20 per cent spiegel is the only one of which there is any doubt of a perfect decarbonisation. The differences, however, are not such as invalidate the process for technical purposes, nor is the alloy one of those to which direct processes only are applicable.

The metallic molybdenum volatilises, leaving only such impurities as SiO_2, WO_3, &c.

The tungsten-nickel alloy has every appearance of being fully oxidised when drawn from the furnace, and repeated ignition yields no more CO_2; but ignition of the residue, or direct ignition of the sample with PbO_2 or Pb_3O_4 indicate a considerably higher percentage. It is noteworthy that ignition with either CuO or $PbCrO_4$ gives lower

figures than ignition with PbO_2 unless a very high temperature is used.

Ferro-manganese, ferro-chromium, chrome-nickel, and metallic chromium resist, with more or less completeness, all attempts to oxidise them without a reagent. Ferro-manganese. however, is not refractory in the sense that metallic chromium or ferro-chromium are. It is not plain why simple ignition decarbonises ferro-manganese so incompletely, unless it be that the hard-gritted surface protects the centre portions from oxidation. It is noteworthy that it decarbonises completely when mixed with such of the metallic oxides as have little or no effect on the chrome alloys. For example, 2 grms. intermixed with 2 grms. of ignited zinc oxide yielded—

6·98 instead of 7·01 per cent carbon
and 6·68 „ 6·63 „ „

Metallic zinc had evidently been formed; there were tufts of crystalline ZnO on the edges of the boat.

Beyond greater simplicity, the advantages of combustion without reagents are:—As there is generally no fusion, the same boat is available time after time; there is no reagent blank to be ascertained, and allowed for; and, where strong reducing agents, like silicon and manganese, are present, no metallic beads (Cu or Pb), with the possibility of occluding some of the sample, can occur.

The choice of zinc oxide in particular cases rests on the facts that it is quite free from blank after ignition in the muffle, and any of the oxide reduced to metal is so readily re-oxidised that no metallic beads can vitiate the assay.

PREPARATION OF METALLIC TELLURIUM.*

By VICTOR LENHER.

Early in 1898 a study was begun on tellurium. The material at hand on which the research was to be made consisted of the so-called electrolytic copper slimes furnished by the Baltimore Electric Refining Company. The first step was naturally to extract the tellurium from these residues, then to work up the metal obtained into such derivatives as might be found convenient for study. The first method employed consisted in the treatment of the residues with concentrated hydrochloric acid; chlorides of the soluble elements were formed, while a highly siliceous residue remained insoluble. The resulting solution was poured into water, when the oxide of tellurium was precipitated along with more or less antimony oxychloride. This precipitate was again dissolved in hydrochloric acid and sulphur dioxide conducted into the solution, when tellurium was precipitated in a finely divided condition.

A process similar to the above is at present the general way by which the metal is obtained. The German tellurium which is on the market is obtained by treatment of the ore with aqua regia and the subsequent precipitation from a hydrochloric acid solution by sulphur dioxide.

A number of methods for obtaining metal are known, but most depend on the precipitation of the tellurium from a hydrochloric acid solution by sulphur dioxide.

Berzelius fused the naturally recurring tellurides with a mixture of oil and soda or potash, or with potassium tartrate. He obtained purple potassium telluride, which, subjected to the moderate oxidation of a current of air, yielded tellurium according to the equation $K_2Te+O+H_2O=2KOH+Te$.

Berthier treats nagyagite, which is a combination of gold telluride and lead sulphide, with hydrochloric acid; hydrogen sulphide is evolved, lead, antimony, &c., pass into solution, while the gold telluride is unattacked. The latter is treated with nitric acid, which dissolves the tellurium away from the gold. The tellurium is brought into hydrochloric acid solution by evaporation with hydrochloric acid, and the tellurium precipitated by sulphur dioxide. Petzite is fused with a mixture of sodium carbonate and potassium nitrate. He extracts the tellurate with water, treats with hydrochloric acid to reduce to tellurite, after which he precipitates with iron. Berzelius extracts in a similar manner, but reduces his tellurate to telluride by fusion with carbon.

Schroetter treats tellurides with either hot concentrated sulphuric acid or aqua regia. The gold is precipitated with ferrous sulphate or oxalic acid and the tellurium by zinc. He re-precipitates the tellurium from a hydrochloric acid solution by sulphur dioxide or an alkaline sulphite. Another method of extraction is to treat the ore with chlorine gas, when tellurium chloride is formed and distils from the non-volatile portion. The tellurium is subsequently precipitated from hydrochloric acid solution by sulphur dioxide. The method doubtless owes its origin to Berzelius. It has also been suggested to prepare tellurium by precipitation with the electric current.

In 1870 Stolba called attention to the precipitation of tellurium from its alkaline solutions by means of grape-sugar; later, Kastner found that it was completely precipitated by grape or invert sugar. But after those two papers the sugar precipitation has been very little heard of; it seemed as though it should be an ideal means of producing metal from the electrolytic copper residues, as the tellurium appears as a waste product and is already in alkaline solution.

A series of experiments was made to attempt the extraction of tellurium by different methods. The first thing to do was to prepare tellurium by a well-known method and obtain a product that it would be possible to use in comparison. To this end, 5 pounds of residues were treated with concentrated hydrochloric acid (sp. gr. 1·20). An amber-yellow liquid was obtained, and a residue which was highly siliceous. It is possible to obtain a perfectly clear solution by filtration through asbestos wool, using a suction-pump. When sulphur dioxide is brought into contact with such a strong acid solution, selenium should be precipitated free from tellurium according to Keller (*Journ. Amer. Chem. Soc.*, xix., 773). When sulphur dioxide was introduced into this solution a red precipitate formed, showing selenium was just precipitated. It appeared to darken, however, when the liquid was saturated. On boiling, the precipitate agglomerated into a mass which much resembled selenium, but, on separating it by means of potassium cyanide, it was found to consist of 3·2 grms. of selenium and 28·3 grms. of tellurium. This seems to indicate that tellurium and selenium cannot be perfectly separated by sulphur dioxide in strong hydrochloric acid solution. The filtrate from the strongly acid solution was diluted with water, and more sulphur dioxide passed through the liquid, when the rest of the tellurium was precipitated. The lump of metal which was formed by fusion was finely powdered and fused with potassium cyanide; purple telluride of potassium was formed. The solution was filtered and a current of air passed through. The tellurium which was formed was fused, then distilled in hydrogen gas. This material was considered pure. When treated with aqua regia, dioxide is formed and may be obtained by evaporation. Tellurium oxide is completely volatile at a low temperature in hydrochloric acid gas, no residue remaining. Pure tellurium is likewise completely volatile when heated in hydrogen.

Since it seems most natural to prepare a metal from its oxide, the tellurium oxide was subjected to a number of reduction tests. In recent years, metallic magnesium and metallic aluminum have shown themselves to be of great value as reducing agents. When metallic aluminum and tellurium oxide are heated together in a crucible a violent reaction takes place, tellurium is formed, but immediately unites with the aluminum, forming aluminum telluride. Metallic magnesium, even in very coarse condition, when heated with tellurium oxide, gives a very explosive reaction.

* Contribution from the Havemeyer Laboratories of Columbia University. From *The Journal of the American Chemical Society*, Vol. xxi., No. 4.

So very energetic is the action that it has not been possible to collect any of the products formed.

When dry glucose is heated with the oxide, a coke is formed which becomes coated with the metal and is difficult to fuse into a button. The same may be said of a dry fusion of asphaltum with the oxide. Ignition of the oxide with dry oxalic acid does, however, give metal readily, and fusion into a globule is an easy matter.

The next series of experiments was made with the sugars. Tellurium oxide was dissolved in potassium hydrate, cane-sugar was added, and the solution warmed. When a saturated solution is used, purple telluride is formed, but boiling in contact with the air causes a rapid separation of black tellurium. After washing with water the precipitate can be dried and fused into a mass.

From a solution of an alkaline tellurite, glucose precipitates black elementary tellurium. No intermediate formation of telluride could be noticed as with cane-sugar. Pure white anhydrous grape-sugar was dissolved in water and added to a warm solution of the alkaline tellurite. Tellurium was precipitated in elementary form. During the process of washing, which always followed the precipitation, it was invariably noticed that grape-sugar was much more difficult to remove from the finely divided tellurium than any of the other sugars.

From these experiments it seemed natural to conclude that reducing sugars will give a very practical method for the preparation of metallic tellurium. Tellurium obtained by this method is completely volatile in hydrogen gas, and its oxide is likewise volatile in hydrochloric acid gas.

Thanks to the kindness of Mr. Walker, of the Baltimore Electric Refining Company, who furnished the residues, and to Prof. P. de P. Ricketts, who in so many ways made the work possible, the author has been enabled to prepare a bar* of tellurium by fusion of the finely divided material obtained by reduction of tellurium in alkaline solution by means of sugar.

Among other experiments, I decided last year, while working on selenium, that as soon as I could obtain satisfactory compounds I should determine the atomic mass of tellurium by precipitation with hydroxylamine. As my work thus far on tellurium, which was begun with Prof. E. F. Smith at the University of Pennsylvania, and is now going on in this Institution, had for one of its objects the determination of the atomic mass of tellurium by means of the hydroxylamine precipitation, it seems quite proper that I should continue it, notwithstanding the fact that Prof. Jannasch, of the University of Heidelberg, announced in the *Berichte* for October, 1898, that he is engaged in work on the same line.

* This bar was exhibited before the American Chemical Society at New York, and weighed four pounds.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 1st, 1899.

Professor Thorpe, F.R.S., President, in the Chair.

(Concluded from p. 82).

14. "*On the Condensation of Formaldehyde with Ethyl Malonate and on the Synthesis of Pentamethylenetricarboxylic Acid.*" By J. Frank Bottomley and W. H. Perkin, jun.

In a previous communication by Haworth and Perkin (*Trans.*, 1898, lxxiii., 330), it was shown that the following substances are formed, either directly or indirectly, by the condensation of formaldehyde with ethyl malonate.

1. *Ethyl methylenemalonate*, $CH_2:C(CO_2Et)_2$, and two polymerides called respectively ethyl *para*-methylenemalonate and ethyl *meta*-methylenemalonate. 2. *Ethyl propanetetracarboxylate*, $(CO_2Et)_2CH\cdot CH_2\cdot CH(CO_2Et)_2$. 3. *Ethyl tetramethylenetetracarboxylate*,—

$$(CO_2Et)_2C<\begin{matrix}CH_2\\CH_2\end{matrix}>C(CO_2Et)_2.$$

In the present paper, the authors show that, under certain conditions, *ethyl pentanehexacarboxylate*, $(CO_2Et)_2CH\cdot CH_2\cdot C(CO_2Et)_2\cdot CH_2\cdot CH(CO_2Et)_2$, is formed by the condensation of 3 mols. of ethyl malonate with 2 mols. of formaldehyde. This new ester is crystalline, melts at 53—55°, and, when digested with baryta, is decomposed with formation of an acid which is evidently propanetetracarboxylic acid, $(CO_2H)_2CH\cdot CH_2\cdot CH(CO_2H)_2$, since on heating at 240° it yields glutaric acid.

Pentanetricarboxylic acid,—

$$CO_2H\cdot CH_2\cdot CH_2\cdot CH(CO_2H)\cdot CH_2\cdot CH_2\cdot CO_2H,$$

is formed when ethyl hexacarboxylate is hydrolysed with dilute hydrochloric acid, and the resulting acid heated at 200°; it melts at 113—114°, and has already been described by Emery (*Ber.*, 1891, xxiv., 284). When ethyl pentanehexacarboxylate is heated with sodium ethylate and methylene iodide, it yields ethyl paramethylenemalonate—a remarkable decomposition, which is discussed in the paper.

Ethyl pentamethylenehexacarboxylate,—

$$\begin{matrix}CH_2\text{—}C(CO_2Et)_2\\|\quad\quad|\\(CO_2Et)_2C\cdot CH_2\cdot C(CO_2Et)_2\end{matrix},$$

is formed when the disodium compound of ethyl pentanehexacarboxylate is treated with bromine, and when hydrolysed yields *pentamethylene hexacarboxylic acid* (m. p. 212°). By the elimination of carbon dioxide from this acid, the *cis*- and *trans*-modifications of *pentamethylenetricarboxylic acid*,—

$$\begin{matrix}CH_2\text{——}CH\cdot CO_2H\\|\quad\quad\quad|\\CO_2H\cdot CH\cdot CH_2\cdot CH\cdot CO_2H\end{matrix},$$

have been prepared; the former melting at 146—148°, the latter at 127—130°. *Anhydro*-cis-*pentamethylenetricarboxylic acid*,—

$$\begin{matrix}CH_2\text{——}CH\cdot CO\\|\quad\quad\quad|\quad\quad>O,\\CO_2H\cdot CH\cdot CH_2\cdot CH\cdot CO\end{matrix}$$

obtained by heating the *cis*-acid with acetic anhydride, melts at 215—217°, and when treated with water it is again converted into the *cis*-acid.

Experiments are also described on the distillation of the *para*- and *meta*-polymerides of ethyl methylenemalonate, and on the hydrolysis of these polymerides and of ethyl methylenemalonate itself.

15. "*The Volumetric Estimation of Potassium.*" By R. H. Adie, M.A., and T. B. Wood, M.A.

The method proposed by the authors is to precipitate the potassium as cobaltinitrite (of which the formula is usually given as $K_6Co_2(NO_2)_{12},3H_2O$) and to titrate the nitrite with a standard solution of potassium permanganate in acid solution. The experiments of the authors so far seem to show that there are three, instead of two, nitrite groups for each atom of potassium, and that the precipitate they obtain is probably an acid salt, as the ratio of the cobalt to the nitrite is that indicated by the above formula.

The method is as follows:—The solution containing the potassium to be estimated is freed from other bases as far as possible, by means of sodium carbonate, then concentrated if the solution is at all weak, acidified with acetic acid, and excess of a solution of sodium cobaltonitrite added. The mixture is allowed to stand for twenty-four hours, the precipitate collected on a Gooch filter, washed several times with 10 per cent acetic acid, and finally once with water. The asbestos filter and the precipitate are now transferred to a beaker and boiled with a dilute solution of soda, filtered, and made up to 100 c.c.

Of this solution 20 c.c. are taken, acidified with dilute sulphuric acid, and rapidly titrated with the permanganate solution. A second fraction of 20 c.c. is taken, and to this the amount just found of permanganate solution is added, then acidified, and the end-point again determined. In one estimation, the volumes required were 16·3 and 16·4 respectively. The end of the reaction is more easily obtained if the permanganate is added to the nitrite solution in excess and the excess determined by potassium iodide and thiosulphate solution.

On testing the method with pure potassium salts, it was found that the results, whilst very concordant amongst themselves, gave results too high in almost exactly the proportion 3 : 2; 1 c.c. of the permanganate solution used was in this way found to correspond only to 0·0077 grm. of K_2O instead of to 0·01175, the value calculated from the formula given above. This seems to indicate for the precipitated salt some formula such as $K_2HCo(NO_2)_6$.

As examples of the accuracy of the method the following analyses are given :—

I. Kainite—

	I.	II.	Mean.
K_2O per cent ..	12·22	11·94	12·13
By platinochloride method—			
K_2O per cent ..	11·98	12·07	12·03

II. Commercial potassium sulphate—

K_2O per cent ..	50·05	50·82	50·44
By platinochloride method—			
K_2O per cent ..			50·80

III. Soil—

	I.	II.	III.	IV.	Mean.
K_2O per cent	1·09	1·20	1·06	1·05	1·10
By platinochloride method—					
	1·11	1·25	0·98	0·97	1·08

The authors are engaged in making a more complete investigation of the composition of the precipitated cobaltinitrite and its decomposition by alkalis, as well as the application of the method to the determination of cobalt.

16. "*On the Action of Aluminium Chloride on Camphoric Anhydride.*" III. By F. H. LEES and W. H. PERKIN, jun.

The authors have already published two notices of their work on the action of aluminium chloride on camphoric anhydride (*Proc.*, 1898, xiv., 111; and 1899, xv., 23), and in spite of this, M. Blanc (*C. R.*, 1899, cxxix., 1019; *Chem. Centr.*, 1900, i., 199) has lately undertaken an investigation of the new substances formed in this reaction which were first obtained by the authors, the examination of which was expressly reserved by them. The authors are therefore compelled to publish the results they have obtained, although the investigation is still incomplete.

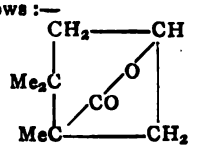
When camphoric anhydride reacts with aluminium chloride at ordinary temperatures, the principal product is *isolauronolic acid*, $C_9H_{14}O_2$, but there is also formed a lactone, $C_9H_{14}O_2$, called by the authors *ψ-campholactone*. This lactone, on hydrolysis with alkalis, yields two hydroxy-acids, $C_9H_{16}O_3$, which melt at 113° and 161° respectively (the melting-points 109° and 152° previously given are too low).

When dissolved in a solution of hydrogen bromide in glacial acetic acid, ψ-campholactone is converted into a mixture of bromo-acids, $C_9H_{15}BrO_2$. One of these is liquid, but the other is a solid melting at 128—130° with decomposition, which when boiled with glacial acetic acid loses hydrogen bromide with formation of an unsaturated acid, $C_9H_{14}O_2$ (m. p. 73°).

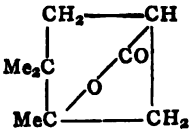
Attempts to reduce the solid bromo-acid with zinc dust and acetic acid led to the formation of a new lactone, $C_9H_{14}O_2$, which melts at 44°, and when hydrolysed with baryta water is converted into the same hydroxy acid, $C_9H_{16}O_3$ (m. p. 113°), which is obtained by the hydrolysis of ψ-campholactone.

Probably ψ campholactone and this new lactone are stereoisomeric, and the same remark applies to the two hydroxy-acids melting at 113° and 161°.

It was stated in one of the previous communications (*Proc.*, 1899, xv., 23) that, when ψ-campholactone is treated with phosphorus pentabromide and the product poured into methyl alcohol, *methyl bromo-dihydro-ψ-lauronolate* is obtained, and that this methyl ester, on hydrolysis, yields an oily unsaturated acid, $C_9H_{14}O_2$, called *ψ-lauronolic acid*. Since the publication of these results, it has been found that this oily acid, on standing for some weeks, deposits crystals which melt at about 69°, and it is evident that the oily acid is a mixture of two isomeric acids, $C_9H_{14}O_2$, which again are probably stereoisomeric. It was also suggested (*Proc.*, 1899, xv., 24) that campholactone and ψ campholactone are possibly closely related, and that this relationship might be represented as follows :—

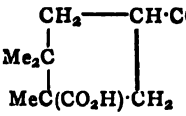


Campholactone.



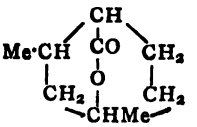
ψ-Campholactone.

These expressions being based on the formula—



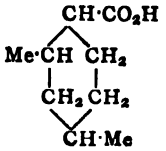
which is one of those suggested by Perkin as possibly representing the constitution of camphoric acid.

When, however, it is remembered that ψ-campholactone dissolved in concentrated sulphuric acid, is almost quantitatively converted into xylic acid at 90°, and that, as has since been found, ψ-lauronolic acid yields the same acid on prolonged treatment with sulphuric acid at ordinary temperatures, it seems possible that ψ-campholactone may after all be the lactone of *hydroxyhexahydroxylic acid*,—



It will be very difficult to prove whether ψ-campholactone is a 5 or 6 ring derivative, since experiments which were made on a considerable scale, with the object of reducing ψ-lauronolic acid—in which case either dihydroisolauronolic acid or hexahydroxylic acid should have resulted and could have been identified—failed owing to the fact that ψ-lauronolic acid is not appreciably attacked even by repeated and prolonged treatment with amyl alcohol and sodium.

When a chloroform solution of camphoric anhydride is left for some weeks in contact with aluminium chloride, the ψ-campholactone remains unchanged, but the isolauronolic acid almost entirely disappears, and in its place two saturated acids, $C_9H_{16}O_2$, are found; these have now been separated partly by freezing and partly by means of their crystalline amides. One of these acids is solid, melts at 77°, and is identical with the *hexahydroxylic acid*,—



which Bentley and Perkin (*Trans.*, 1897, lxxi., 173) obtained by the reduction of xylic acid. The other acid, $C_9H_{16}O_2$, is an oil and it is probable that this and the solid acid represent the *cis-* and *trans*-modifications of hexahydroxylic acid. When the acid melting at 77° is treated with bromine and subsequently with alcoholic potash, it yields a solid acid, $C_9H_{14}O_2$ (m. p. 107°), which is identical with the *tetrahydroxylic acid* prepared by Bentley and Perkin (*loc. cit.*).

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 5, January 29, 1900.

Study of the Rays of Radium.—H. Becquerel.—The portions of the radium rays which are deviated by the magnetic field lend themselves to various experiments, from the study of which the author has found—(1). The influence exerted by air on the velocity of propagation of the rays in question, by making measurements in a vacuum. However, he observed no appreciable difference between vacuum and air measurement. (2). The identification of rays emitted by variously active radiferous salts. (3). The form of the trajectory of the rays in a uniform magnetic field. (4). The dispersion in the magnetic field. (5). The existence of electrostatic charges in the presence of very minute material masses.

Molecular Volumes of some Camphor Derivatives.—A. Haller and P. Th. Muller.—The authors' experiments show that the decrease of volume corresponding to the actual addition of the two radicles to form camphor is about 23 c.c. per litre, and is less for toluenic solutions, which are nearly a quarter normal. This number, which is still considered provisional, allows nevertheless of fixing in a sufficiently accurate degree the molecular weight of camphor compounds by the method of densities. It allows also the disclosure of the existence and production of new closed chains in the derived molecule of camphor.

Retention of Silver Chloride by Mercurous Chloroamide.—F. Leteur.—To estimate silver in presence of mercury, the usual plan is to treat the mixture of the two chlorides with aqueous ammonia. The mercurous chlorides is transformed into mercurous chloroamide, $NHg_2H_2.Cl$, and the silver chloride is dissolved. This simple method succeeds when the latter substance exists in a tolerable quantity in the mixture, but it is insufficient when the contrary is the case, the complex mercurous compound retaining always some silver chloride. The author having examined the nature and quantity of this retention suggests the following method of procedure:—The residue from the treatment of the mixed chlorides with aqueous ammonia is digested in the cold with concentrated nitric acid, to which is added sufficient hydrochloric acid to transform the chloride of mercuroso-ammonium into mercuric chloride. In the case of the presence of silver, there remains a white residue, which is not blackened by aqueous ammonia and is totally soluble in this reagent. If the residue does not present this character another treatment must be performed.

Action of Copper on Acetylene. Formation of a Highly Condensed Hydrocarbide—Cuprene.—Paul Sabatier and J. B. Senderens.—The author's experiments on the action of a mixture of excess of hydrogene and acetylene on various metals shows that iron and cobalt behave in the same manner as nickel; the action of the latter having been discussed in a former paper. Copper, however, appears to exert a special reaction on acetylene alone. The resulting product is a yellow solid, which is apparently built up of a mass of very fine filaments. It burns quickly, giving out an aromatic odour, and leaving a slight black residue of cupric oxide. It is evidently a hydrogen carbide. Analysis shows that the relation between the carbon and hydrogen is 14 : 1. On account of its method of formation the name cuprene is proposed, the formula being C_7H_6. When heated to above 400° it decomposes, evolving complex products, and leaving a solid residue of carbon.

Acidimetry of Polybasic Organic Acids.—A. Astruc.—This paper contains an account of further experiments on the action of polybasic organic acids on various indicators, and is a continuation of the author's work with M. Henri Imbert, which was published in a previous paper.

Isopyromucic Acid.—L. T. Simon.—Pyromucic acid was discovered as one of the products of distillation of mucic acid, being produced according to the equation, $C_6H_{10}O_8 = C_5H_4O_3 + CO_2 + 3H_2O$. In 1873 Limpricht, whilst repeating this distillation, isolated as a by-product an acid which he considered as an isomer of the preceding acid. The author repeats this experiment and succeeds in preparing the latter acid exclusively. The isopyromucic acid thus prepared is white when pure, very soluble in alcohol, ether, and water, and but slightly soluble in carbon disulphide.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Water Analyses.—(Reply to N. H.).—The quantity of $CaSO_4$ present is excessive, and would in itself be sufficient to render the water undesirable for drinking or domestic purposes; 30 parts of total hardness and 50 parts of total solids per 100,000 is the maximum that a potable water should contain.—W. A. Jenkin, Rio Tinto Laboratory, Spain.

MEETINGS FOR THE WEEK.

Tuesday, 27th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
— Society of Arts, 4.30. "Agricultural Education in Greater Britain," by R. Hedger Wallace.
Wednesday, 28th.—Society of Arts, 8. "Pneumatic Dispatch," by Prof. Charles A. Carus Wilson, M.A.
Thursday, March 1st.—Royal Institution, 3. "Recent Excavations at the Argive Heræum (in Greece)," by Charles Waldstein, Litt.D., Ph.D., L.H.D.
— Chemical, 8. "Pilocarpine and the Alkaloids of Jaborandi Leaves," by H. A. D. Jowett, D.Sc. "Isomeric partially Racemic Salts containing Pentavalent Nitrogen" (Parts I.—VIII.), by F. S. Kipping, D.Sc., F.R.S. "New Synthesis of Indene," by F. S. Kipping, D.Sc., F.R.S., and H. Hall. "Potassium Nitrito-hydroximidosulphates and the Non-existence of Dihydroxylamine Derivatives" and "Identification and Constitution of Fremy's 'Sulphazotised Salts of Potassium,'" by E. Divers, D.Sc., F.R.S., and T. Haga, D.Sc. "Some Acids obtained from α-Dibromocamphor," by A. Lapworth and E. M. Chapman.
Friday, 2nd.—Royal Institution, 9. "Malaria and Mosquitos," by Major Ronald Ross, D.P.H., M.R.C.S.
— Physical. (Special Meeting in the Physical Laboratory of University College. The Laboratory will be open and tea served at 4.30). "The Relative Rates of Effusion of Argon, Helium, and some other Gases," by Dr. F. G. Donnan. "On the Distillation of Liquid Air and the Composition of the Gaseous and Liquid Phases," by E. C. C. Baly. "The Reversibility of Galvanic Cells," by T. S. Moore. "On the Damping of Galvanometer Needles," by M. Solomon.
Saturday, 3rd.—Royal Institution, 3. "Polarised Light." by The Rt. Hon. Lord Rayleigh, M.A., D.C.L., &c.

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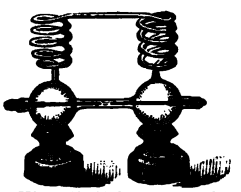

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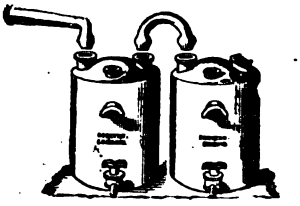

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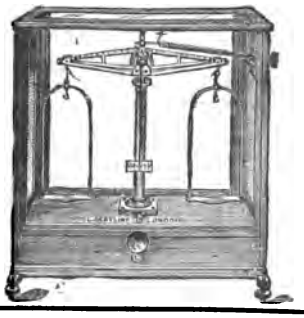

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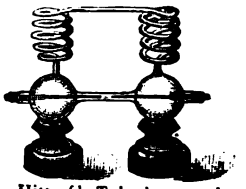

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NOTE ON THE DETERMINATION OF NITRIC NITROGEN BY SCHLÖSING'S METHOD.

By C. DAVIDSON.

THE distinctive feature of de Koninck's apparatus (*Zeit. Anal. Chem.*, xxxiii., 200), abstracted in *Journal of Chemical Society*, 1894, ii., 296) is the capillary delivery tube which dips under mercury, and is longer than a barometer-tube, so that the functions of the troublesome stopcocks or slips, usual in apparatus for this purpose, are automatically performed. The following slight modification of de Koninck's apparatus, besides practically eliminating the error due to the solubility of nitric oxide in water, has proved very convenient in actual use during the last two years.

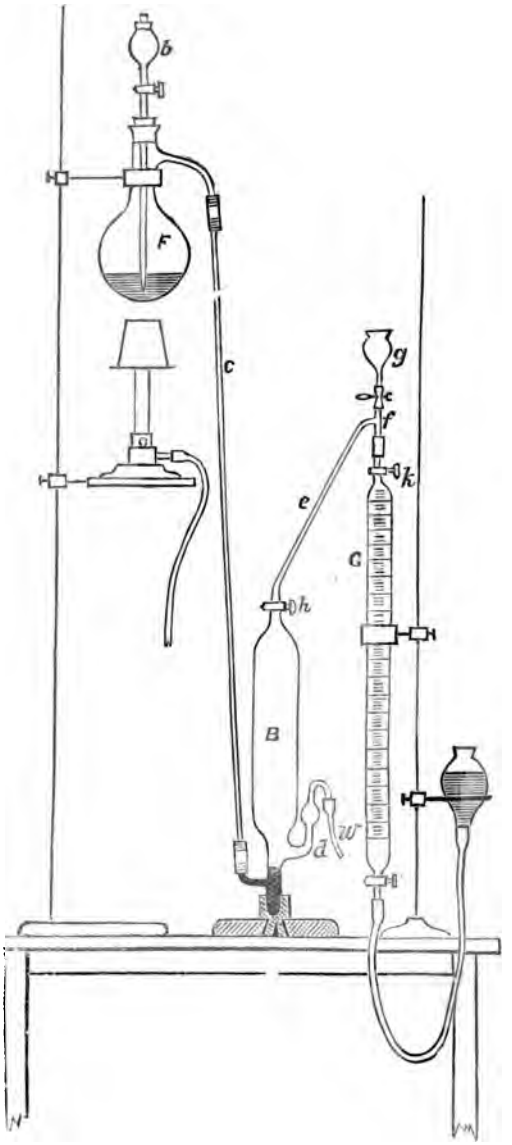
The principle of the apparatus is to substitute for de Koninck's mercury trough and large cylinder of water a modified Schiff's nitrometer, which is rather more convenient to handle, although initially more troublesome to make.

Apparatus.—The stoppered funnel *b* has a capacity of about 50 c.c., and should if possible have a capillary stem. F is a distilling flask of 250 c.c. capacity, with its side-tube bent almost perpendicularly. The tube *c* is of fine bore—in my own apparatus about 0·2 c.m. diameter—and of such length that the difference of level of its two ends is about 80 c.m. Joints of *thick-walled* rubber tubing have proved quite safe if well corded. The tube B is really a modified Schiff's nitrometer; the capacity of the wider part is about 200 c.c.; the narrower part at the bottom is about 1·5 c.m. in bore and 6 c.m. long between the two side-tubes. The tube *d* is blown on so that its upper-side slopes continually *upwards* towards B, while its underside is at least level. The tubes *e* and *f*, forming a T which connects B on the one hand to a gas-burette (capacity 100 c.c.), and on the other to a small bulb, *g* (whose use is obvious), are both capillary. The lower part of B is filled to within a c.m. of *d* with mercury. The rubber tube *w* communicates with a tubulated bottle, W (not shown in the sketch), containing air-free water.

Procedure.—Forty c.c. of a solution of $FeCl_2$, containing 20 per cent Fe (made from wrought-iron nails and HClAq, air being excluded by a layer of light petroleum), together with an equal bulk of HClAq of sp. gr. 1·10, are placed in the flask F. The stem of the funnel *h* is filled with water; and the reservoir W having been filled with (warm) water from which the air has been expelled by boiling, the tube B and the capillary *e* are filled with water therefrom. The burette G should not be connected up as yet.

Heat is applied to F by means of a small Bunsen-flame whose tip just touches the flask; and the heating is continued until all the air has been boiled out into B, whence it is removed, from time to time if necessary, through *e f*, by keeping W high and opening the stopcock of B. When air ceases to come over, the Bunsen is removed (or lowered on its rest until the mercury in *c* is just kept stationary), B is completely filled with water from W, the overflow going out through *f*; the gas-burette, G, charged with air-free 7 per cent NaOHAq, is joined on at *f*; any air-bells remaining in *f* are removed by raising the burette-reservoir and opening the stopcock and clip. Enough soda solution should be allowed to flow through to half-fill the bulb *g*; it will be useful afterwards.

Such a quantity of the solution of the substance to be operated on as corresponds to about 0·2 grm. of $NaNO_3$ is placed in the funnel *h*, and carefully run into F. The funnel is rinsed out three times with 10 c.c. of HClAq, of sp. gr. 1·10, care being taken, of course, to keep out air; W is placed high and the burette-reservoir low, and heat is cautiously applied to F. If the mercury in *c* is at this point more than half-way up, the heating must be very gentle at first (a Bunsen-flame an inch long, and sheltered by a funnel, should be placed with its tip about 2 inches below the flask, to begin with), but as the mercury falls the heat may be increased, until when the liquid is boiling



under atmospheric pressure no special care is necessary. By simply opening the two stopcocks, *h* and *k*, the NO is transferred from time to time to the gas-burette (the lower stopcock of which is of course open all this time).

When all the NO has been boiled out of F, it is swept into the burette finally by raising W sufficiently and opening *h* and *k*; if any small bubble sticks at the join of the T, it is removed by closing *h*, keeping *k* open, and opening the clip of *g*, which was half-filled with soda-solution at the start. When the bubble has gone into

the burette, *k* is closed and then the clip; the burette is removed, cooled suitably, and the volume of the gas noted, together with the temperature and barometric height at the time. During the cooling it is well to close the lower stopcock of the burette, and invert the latter once or twice, in order to keep fresh soda in contact with the gas.

"Bunsen's 'Gasometrische Methoden' (second edition) contains a table giving the tensions of vapour of water given off by such a caustic soda. If this table is not at hand, subtract from the tension of pure water the following correction:—

At 10° to 15° C. .. .. 1·1 m.m.
At 15° to 25° C. .. .. 1·4 m.m. } Kreusler.

And the result is right to within ± 0·2 m.m. of mercury."—Dittmar, "Quantitative Analysis," p. 89.

One need not be painfully exact about this correction of the vapour tension, for the error caused by neglecting to make the above subtraction is in the worst case stated less than + 0·16 c.c. per hundred of NO.

As to the error caused by the solubility of the gas in water, it was found that the substitution of a reservoir of *boiling* water (kept boiling during the experiment) for W had no effect on the result. The error in question must therefore be very small.

A determination, including the preliminary boiling-out of the air, can easily be performed within an hour; and (as the $FeCl_2$ used suffices for over 800 c.c. of NO) if a number of determinations be performed consecutively, the average time of a determination is not much over half-an-hour.

The apparatus may very conveniently be mounted on a low stool, about 18 inches high, having a retort-rod 3 feet long screwed into it, and preferably provided with casters on two of its legs, so that it may be wheeled into a corner when no longer required.

In concluding, the author thinks it not out of place to utter a word of warning with regard to the practice, prevalent in some quarters, of attempting to compensate for inexact methods of analysis by comparing the results of two "parallel" experiments — one on an unknown substance, and the other on a standard whose content of the constituent sought is assumed to be known. Such methods are at best of but limited application. In many cases the purity of the standard may be doubted, while in others the precautions necessary to ensure that the parallel experiments are really being worked under exactly similar conditions are as laborious as those which would have to be taken in order to eliminate the errors whose effects are sought to be compensated.

37, Herriet Street, Pollokshields,
Glasgow.

ON THE

DEGREE OF PULVERISATION OF THE RAW MATERIALS IN THE MANUFACTURE OF CARBIDE OF CALCIUM.

By B. CARLSON.

There is still a good deal of difference of opinion as to the degree of pulverisation necessary for the raw materials in the manufacture of carbide of calcium. If we obtain the same returns with a mixture of finely powdered lime and coke as with a coarsely powdered one, the second is evidently to be preferred from the economic point of view.

Now the heat necessary for the reaction may be divided into the heat of the reaction proper, and the heat necessary to bring the raw materials to the necessary temperature. Neither the specific heat, nor the heat of fusion of solid bodies, depends on their degree of fineness; it is the same for the heat of combination which is determined when the temperature is reached. A finely powdered material and an intimate mixture would only be necessary if:—1. It was difficult to reach the temperature of the formation of carbide in the electric furnace; 2. If neither one nor the other of the raw materials, coke and lime, were liquid at the temperature obtained in the furnace; and 3. If no liquefied body was produced in which the raw materials could dissolve. In such a case, it would be necessary to bring into the zone of formation of the carbide as intimate a mixture as possible. Now, neither one nor the other of these conditions are fulfilled in this industry. The temperature at which the carbide is produced is decidedly lower than that of the electric furnace, and the lime also melts well below this temperature. The product of the reaction is, further, a solvent of lime and of carbon.

The process of the formation of carbide of calcium should be the following:—The lime should melt first and the coke dissolve in it, with the production of carbide of calcium. This reaction is absolutely analogous to that which takes place between iron and dilute sulphuric acid, for example. A solid body dissolves in a liquid with the evolution of gas. Under these circumstances the degree of subdivision of the material is absolutely unimportant.

So much for the theoretical point of view; we will now show that it is confirmed by practice.

If the mixture of lime and coke is in the state of powder, the carbonic oxide gas is unable to escape freely, and an empty space is formed round the igneous zone; the sides of this space are quickly brought to a high temperature by the radiation from the arc, and a species of lining of carbide is produced,—that is to say, a short circuit is formed between the electrodes. The pellicle thus formed has a certain resistance, and it is only after the lapse of a certain time that it suddenly explodes under the pressure of the carbonic oxide continually being formed. The gases make a passage through the charge, carrying with them a considerable quantity of fine powder; other explosions, arising from the mixture of carbonic oxide and air, and resulting from the furnace not being air-tight, are easy to prevent. The passages formed by the gases last for a certain time; their sides, formed of slightly conducting materials, rapidly take the temperature of the carbonic oxide, and this latter escapes from the furnace at approximately the temperature of the arc. It has been said that the temperature of the carbonic oxide ought to be considerably lower, since, being a product of solid coke and lime, it should absorb the heat in becoming gaseous. This would be true if, as soon as it was formed, the gas was removed from the action of the arc; but as this is in no way the case, it rapidly acquires a temperature near that of the arc. As soon as the passages of which we have spoken become obstructed, the production of a bulb of gas, and the other phenomena described, recommence.

Each time this takes place, a certain quantity of the comparatively cold substance falls into the zone of action of the arc, and causes the resistance to vary. It is for this reason that such large variations of current are met with in most manufactories of carbide. The dynamos also suffer from these variations after a time, and they should be prevented as much as possible. It is of course true that the bad effects can be avoided by having more powerful generators, but that leads to a larger capital outlay on the factory.

In certain factories the bubbles of gas are broken by wooden sticks as soon as they form; but even this remedy is insufficient, and necessitates very exhausting work.

These disadvantages can be entirely removed by substituting, for the powdered mixture, pieces the size of a nut. The gases produced pass freely throughout the mass, which they warm up, and escape from the furnace at a maximum temperature of 1000—1100°: as the temperature of the arc is generally 3000—4000°, the difference represents a considerable quantity of heat given to the raw materials; these latter are further, in this case, completely deprived of water and carbonic acid, and the removal of these impurities increases the return to some

extent. The current passing through the furnace also becomes more constant.

By using a coarsely powdered material we can prevent both the production of dust and the inconveniences which accompany it. The gases traverse the layer of material not yet brought into action, and thus the expense of special apparatus to effect this is avoided.

When the mixture of lime and carbon is finely powdered, the gases carry off much more carbon, which is light, than lime, and an excess of this more expensive material has therefore to be used; further, we can never be sure what the proportions of the mixture are. When the manufacture is interrupted, the residue remaining in the furnace cannot be made use of without a preliminary analysis. This drawback has been remedied by making the residual dust into bricks, but it is more simple only to use the raw materials coarsely broken up. The loss of carbon is then reduced to a minimum, and it is not necessary to use more than the theoretical quantity. The equation $CaO+3C=CaC_2+CO$ shows that 1440 kilos. of raw material are required to obtain 1 ton of carbide. At the "Deutsche Gold-und-Silber-Scheide Anstalt," at Frankfort-on-Maine, 1590 kilos. were used, while at some factories, where the powder was used, as much as 3000 kilos. were required. With coarsely powdered material it is only necessary to take a few precautions, very simple but indispensable for the success of the operation; it may be for this reason that it is so seldom used.

M. Pfleger, in 1897, made some conclusive experiments on this subject. Intermittent manufacture is more advantageous than continuous working, and all inconvenience due to dust in the furnace is got rid of.—*Zeitschrift für Elektrochemie*, vi., p. 325.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JANUARY 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, February 12th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Jan. 1st to Jan. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during January was 2·30 inches, very evenly distributed throughout the month; the average fall is 2·16 inches. This makes an excess of 0·14 inch, or 6·4 per cent on the thirty years' average.

Our bacteriological examinations of 557 samples have given the results recorded in the following table; we have also examined 36 other samples, from special standpipes, &c., making a total of 593 samples:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	621
New River, filtered (mean of 131 samples) ..	10
Thames, unfiltered (mean of 27 samples) ..	2235
Thames-derived water, from the clear-water wells of eight Thames-derived samples (mean of 306 samples)	22
Ditto ditto .. .. highest	292
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 26 samples) ..	694
River Lea, from the East London Company's clear-water well (mean of 41 samples) . ..	7

These results show that during the past month the London water supply has been highly satisfactory, both bacteriologically and chemically.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

ON THE MODIFICATIONS OF KJELDAHL'S METHOD FOR THE ESTIMATION OF NITROGEN.

By A. ATTERBERG.

K. WEDEMEYER has shown that the estimation of nitrogen by Kjeldahl's method can be effected in an hour; this statement caused me to make the following experiments:—

I first of all compared the various modifications of Kjeldahl's method, as they are described in König's book ("Unters. d. Landw. Stoffe," 2nd edition, p. 133), and specially noted the length of time required by the different oxidising agents to obtain complete oxidation and decolouration of the liquid. The material to be oxidised was represented by an earth containing 15 per cent of water and leaving 7 per cent of ash. The following are the results obtained:—

	Minutes.
Method No. 1, with the addition of mercury	45
" " 2, " "	45
" " 3, " " about	55
" " 4, " "	55
" " 5, " "	55
" " 6, without, " "	95
" " 7, " " "	95

The acceleration of the oxidation by the addition of mercury caused me to pursue my investigations in the same direction, and my experiments were carried out on the same sample of earth. I may also mention that all my experiments were carried out in flasks of 250 c.c. capacity, except where the contrary is stated.

	Minutes.	
Gunning's mixture (20 c.c.) without mercury	95	With abundant froth.
The same, with 1 drop of mercury .	55 48 42	Abundant froth at the commencement.
Concentrated sulphuric acid + Hg; with addition of potassic sulphate after complete solution of the earth. Proportion of H_2SO_4 to K_2SO_4 the same as in Gunning's mixture	32 27 26 25	No froth.
The same, without mercury	92	No froth.

The following experiments were made in a flask of 375 c.c. capacity:—

	Minutes.	
Gunning's mixture + Hg	30	Abundant froth.
Concentrated H_2SO_4 + Hg and then K_2SO_4	17	No froth.
Concentrated H_2SO_4 + Hg	65	No froth.

Oxidation thus occurs more rapidly when we add sulphuric acid and mercury, and then, after the solution of the earth, potassic sulphate; it is also accelerated by the use of larger flasks.

The use of mercury requires the addition of potassic sulphide when distilling the ammonia. To do without this addition, I endeavoured to replace the mercury by some other metal.

c.c.		Grms.	Minutes.	
20 H_2SO_4,	without addition, +	18 K_2SO_4	90	
,,	+ 0·3 grm. As_2O_3 +	,,	60	Abundant froth.
,,	+ 0·5 ,, Sb_2O_3 +	,,	70	
,,	+ 1·0 ,, Sn +	,,	60	
,,	+ 1·0 ,, Bi_2O_3 +	,,	70	
,,	+ 1·0 ,, MoO_3 +	,,	38	Froth disappears rapidly.
,,	+ 1·0 ,, MoO_3 +	,,	35	
,,	+ 1·0 ,, MoO_3 +	,,	33	
,,	+ 3·0 ,, MoO_3 +	,,	30	

It thus appears that molybdic acid offers some advantages over mercury; it makes the addition of potassic sulphide unnecessary, and the time occupied by the oxidation is not increased.

But, on the other hand, the molybdic solution forms a considerable amount of froth on the addition of the potassic sulphate. Further, after oxidation, it is of a strong blue colour, which makes the termination of the oxidation difficult to observe with accuracy. It must also be remembered that commercial molybdic acid frequently contains nitrated impurities. It results from this that although offering certain advantages, molybdic acid does not make a very good substitute for mercury.

Kellner's modification gives fairly rapid results. I made the following trials:—

Kellner's mixture (20 c.c.) + Hg..	45 minutes.
	55 ,,
	41 ,,
,, ,, without Hg, with K_2SO_4.	50 ,,
,, ,, ,, ,, ,,	57 ,,

The following experiments were made with the view to find the best proportions between the sulphuric acid and the potassic sulphate:—

			Minutes.	
20 c.c. H_2SO_4 + Hg and	7·5 grms.	K_2SO_4	41	No froth.
,, ,, ,,	10·0 ,,	,,	30	,,
,, ,, ,,	12·5 ,,	,,	32	,,
,, ,, ,,	15·0 ,,	,,	30	,,
,, ,, ,,	17·5 ,,	,,	29	,,
,, ,, ,,	20·0 ,,	,,	26	Rather a lot of froth.
,, ,, ,,	30·0 ,,	,,	45	Very abundant froth.
,, ,, ,,	10·0 ,,	,,	34	Very little froth.
,, ,, ,,	7·5 ,,	,,	40	Hardly any froth.

In the last two experiments the potassic sulphate was added at the commencement of the operation. These figures show that the quantity of this salt to be added varies from 10 to 18 grms.

To sum up, therefore, the best oxidising solution for Kjeldahl's process for the estimation of nitrogen is composed of a mixture of 20 c.c. of concentrated sulphuric acid and from 15 to 20 grms. of potassic sulphate, with the addition of a little mercury.

It is more advantageous to add the potassic sulphate when the solution is complete, but when we have to deal with substances which do not froth it can be added at the commencement of the operation. Under these conditions the liquid becomes colourless after only thirty minutes, but it is advisable to heat it for another quarter of an hour.—*Chemiker Zeitung*, xxii., p. 505.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extra Meeting, February 8th, 1900.

Sir Henry Roscoe, LL.D., F.R.S., Vice-President, in the Chair.

The President, Professor T. E. Thorpe, LL.D., F.R.S., delivered the Victor Meyer Memorial Lecture.

Victor Meyer was born in Berlin on September 8th, 1848, and died in Heidelberg on August 8th, 1897. He was at the time of his death President of the German Chemical Society, and had been an Honorary Foreign Member of the Chemical Society since 1883. As a friend of nearly thirty years' standing, and as one who studied with him under Bunsen, the President had acceded to the request of the Council to record its appreciation of the remarkable services Victor Meyer rendered to the science which he cultivated during the all too short period of his activity with such striking assiduity and success.

Some account was given of Meyer in his student days in Heidelberg, of his early connection with the University Chemical Society, and of his work as one of Bunsen's assistants. In 1868 he entered Baeyer's laboratory at the Gewerbeakademie in Berlin, and began the series of half-dozen investigations which characterised his activity during his three years' stay in the German capital. Special reference was made to his memoir on the constitution of the disubstituted benzenes, and upon his method of introducing a carboxyl group into the molecule of aromatic compounds by the action of sodium formate upon the potassium salt of the aromatic sulpho-acids, and it was shown how the results of his observations led to a revision of the views then held with respect to the orientation of the radicles in the "ortho" and "meta" (salicylic) series.

Meyer's success as a private teacher induced von Baeyer to recommend him to Fehling, who sought assistance, especially in the teaching of modern organic chemistry and in practical work in connection with his duties at the Stuttgart Polytechnic. Although he remained in Stuttgart barely a year, he there made one or two discoveries, especially that of the nitro-compounds of the fatty series, which at once stamped him as an original investigator of a very high order. From Stuttgart he was called to Zürich as the successor of Wislicenus, who had been moved to Würzburg, and, when barely 24 years of age, he was elected Ordinarius and Director of the chemical laboratory of the Polytechnic. The thirteen years of Meyer's stay at Zürich constitute the most fruitful and the most brilliant period of his career; before its close he had brought himself within the foremost rank of contemporary investigators. Some idea of his wonderful power of work, and of the stimulus he gave to others, may be gleaned from the fact that during that period the Zürich Laboratory gave close on 130 papers and memoirs to chemical literature. Special stress was laid upon the elaborate investigation of the nitro-compounds of the alkyl series and their derivatives, which continued to occupy Meyer and his pupils for many years. Reference was made to the discovery of the nitrolic and azaurolic acids, and of the pseudonitroles, and it was pointed out how the characteristic colour reactions afforded by the behaviour of nitrous acid with the primary and secondary nitro-paraffins, and its inability to act upon the tertiary compounds, offered a ready means of distinguishing primary, secondary, and tertiary alcohol radicles. It was shown how the same line of inquiry led to the discovery of the group of the azines and of the ketoximes and aldoximes, and what the influence of these discoveries was upon the stereochemistry of nitrogen.

It was at Zürich that Meyer devised his various methods of determining vapour densities, and it was pointed out

what an important influence the simple and extremely convenient process, known as the air displacement method, has had upon our knowledge of the true molecular weights of substances. Meyer's work on the dissociation of the halogens was especially referred to. Pyrochemical problems, in fact, continued to interest him to the end, and he was quick to take advantage of any hint which seemed to promise the possibility of their solotion. In a lecture before the Naturforscher Versammlung, in Heidelberg, he regretted that the lack of vessels of sufficiently refractory material prevented him from working at the higher limits of temperarure even then attainable. "There can be no doubt," he said, "that new and undreamt of discoveries will manifest themselves—that a new chemistry will disclose itself—when we are furnished with vessels that enable us to work at temperatures at which water can no longer exist, and at which oxyhydrogen gas becomes an uninflammable mixture."

In the autumn of 1882 Meyer was requested to undertake the delivery of the series of University lectures on benzene derivatives which had been interrupted by his friend Weith's death. It was in the course of these lectures that he came upon what is, perhaps, the most brilliant of all his discoveries—that of thiophen. He desired to show his class the indophenin reaction of Baeyer, which, at that time, was held to be indicative of benzene; but, to his astonishment, not a trace of the characteristic blue colour made its appearance, although, as was his wont, he had rehearsed the experiment just prior to the lecture. It happened that his assistant had handed to him a sample of the benzene which had been made in the lecture couree by heating benzoic acid with lime, and at once drew his attention to the fact that the rehearsal had been made with the ordinary laboratory supply, which, of course, was derived from coal-tar. It was soon definitely established that it was only coal-tar benzene that gave the indophenin reaction. Meyer's first idea was that it might be occasioned by an isomeric, a second benzene found in coal-tar; but he quickly ascertained that the reaction was due to some sulphuretted product, and that Baeyer's indophenin was a sulphur compound. He found that coal-tar benzene, after repeated shaking with oil of vitriol, no longer reacted with isatin, and it was among the sulphonic acids so formed that he discovered the reactive substance. This he was at first inclined to call *thianthren*, then *thiophan*, next *thiol*, and lastly *thiophen*, to denote that it was a sulphur-containing substance giving derivatives analogous to those of phenyl. Within about six months of his first observation, he was in a position to show to the Swiss Naturforscher Versammlung, by actual preparations, that its chemistry was hardly less extensive than that of benzene itself. In 1888 he published, in the form of a monograph—"Die Thiophengruppe" (Braunschweig: Vieweg u. Sohn)—an account of its condition at that time, from which it appears that, during the preceding five years, no fewer than 106 contributions to its history had been made from his own laboratory, and some 40 from those of others.

On the death of Hübner, Meyer received a call to Göttingen, which he accepted, and where a new laboratory was to be built for him. Here he continued his pyrochemical work, and his investigation of the thiophen derivatives, and began with Paul Jacobson the admirable "Text-book of Organic Chemistry," which, in the critical selection and arrangement of its material, is unsurpassed. With Auwers, he resumed the study of benzil and its derivatives, which he had begun with Wittenberg and Goldschmidt, and with Münchmeyer he commenced the study of the behaviour of phenylhydrazine towards various groups of oxygen compounds. With Demuth, he undertook the investigation of the sulphuranes. Other notable papers of this period were on isophthalaldehyde; on the negative nature of the phenyl group; on the thio-derivatives of deoxybenzoin and its analogues (desaurins); and with Riecke on the carbon atom and valency.

Stereochemical questions—we owe the phrase to Meyer—occupied much of his thought at this period. His perspicacity and insight are well illustrated in the lecture he gave to the German Chemical Society, in 1890, on the results and aims of stereochemical research. It is of interest to the student as giving a fairly complete historical account of the development of space formulæ, and more especially for the criticism of the work of Baeyer and Wislicenus on the stereochemical formulæ of single-, double-, and treble-linked carbon compounds, and of the stereochemical conceptions of Hantzsch and Werner in the case of nitrogen compounds. To this period belongs also his work on the aromatic nitriles, on tetramethylsuccinic acid, and on the oximes of phenanthraquinone.

Meyer was not destined to remain long in Göttingen. The new laboratory was barely finished when, in 1889, he received a call to Heidelberg, as successor to Bunsen. To Heidelberg accordingly he went with the coveted title of Geheimrath and the promise of a new and enlarged laborarory. Although only 40 years of age, he was now, so far as worldly position was concerned, at the summit of his career. In conjunction with a number of his pupils he began the investigation of the conditions determining both the gradual and the explosive combustion of gaseous mixtures, notably of electrolytic gas, and of the simpler hydrocarbons with oxygen. In the case of mixtures of aliphatic hydrocarbons with equivalent amounts of oxygen, it appeared that the temperature of explosion falls as the number of the carbon atoms in the molecule increases; that it is probably lower for primary than for corresponding secondary hydrocarbons; and is less for hydrocarbons containing a double bond than for those containing only single bonds, and is still less for those containing a triple bond.

In 1892 Meyer and Wachter announced the existence of the iodoso-compounds, a series of aromatic derivatives in which the monovalent group IO replaces hydrogen. The study of these compounds led to the discovery of the remarkable substances known as the iodonium bases—compounds in which two of the valencies of the trivalent iodine atom are satisfied by aromatic radicles, while the third is satisfied in the free base by hydroxyl, and in the salts by an acid radicle. The iodonium bases are readily soluble in water, are strongly alkaline, and in their behaviour, as in that of their salts, show a remarkable similarity with the derivatives of silver, lead, and more particularly thallium.

Reference was also made to the study of the conditions determining the formation and hydrolysis of ethereal salts of aromatic acids, which occupied Meyer, in conjunction with his pupils, and more especially Sudborough, from 1894 up to the year of his death.

Meyer contributed to the literature of chemistry, either alone or in conjunction with his pupils, upwards of 300 memoirs and papers.

As the director of a large chemical laboratory, and as a laboratory teacher, Meyer worthily followed in the footsteps of Bunsen. He was an admirable lecturer, clear, vigorous, and extremely lucid, and his lectures were most popular. As a teacher, he had a wonderful faculty of infusing enthusiasm into his students, His literary ability, combined with his power of exposition, made him an admirable writer of popular science articles. His merits were recognised in every land where science is cultivated, and he was an Honorary Member of many Continental academies. The Royal Society awarded him the Davy Medal in 1891, and in the spring of that year he attended the celebration of the Jubilee of the Chemical Society, and the eloquent words which he uttered in responding to the toast of "Our Foreign Members" will not readily be forgotten by those who attended the banquet on that occasion.

On the motion of Professor Dewar, seconded by Dr. Armstrong, a vote of thanks was passed to the President for his lecture.

Ordinary Meeting, February 15th, 1900.

Professor Thorpe, F.R.S., President, in the Chair.

Messrs. J, Dewhirst, Edgar M. Chapman, and Thomas A. Henry were formally admitted Fellows of the Society.

It was announced that the following changes in the Council were proposed by the Council:—

As *Vice-Presidents*—Professor Divers, M.D., F.R.S., and Dr. T. Stevenson, *vice* Professor Ramsay, F.R.S., and Professor Emerson Reynolds, F.R.S.

As *Ordinary Members of Council*—Dr. Chattaway, Professor Collie, F.R.S., Professor Augustus Dixon, M.D., and Mr. W. J. Pope, *vice* Mr. Bevan, Dr. Moody, Dr. H. F. Morley, and Professor Smithells.

Certificates were read for the first time in favour of Messrs. Alexander William Bain, 2, Muswell Rise, Highgate, N.; Arthur Charles Davis, 3, Vinery Road, Cambridge; Noel Fielding Deerr, 26, South Parade, Chelsea, S.W.; Adolf Liebmann, 10, Marsden Street, Manchester; Frederick Thomas Munton, The Oak House, Winsford, Cheshire; James Wallace Sandford, Adelaide, S.A.; Bertram D. Steele, 40, Herndale Road, London, S.W.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—

Frederick Nolan Baker; Alexander Hutcheon Bennett; Sidney Calvert, M.A., B.Sc.; Oscar Joseph Cole, B.A.; William Francis Cooper, B.A.; Thomas Cuthbert Davison; Harry M. Dawson, B.Sc., Ph.D.; Stephen M. Dixon, B.A.; Alexander Findlay, M.A., B.Sc.; James Foulds; Granville Reginald Gee; Julius Geldard; Robert W. Gray; Alfred William Harvey; Herbert William Hart; Adam Houston; Henry William Hutchin; Henry Jackson, B.A., B.Sc.; Thomas Goode Joyce, B.Sc.; W. C. Robert Kynaston; Arthur Robert Laws; Thomas Ebenezer Mackenzie; William Mair; A. W. Cranbrook Menzies, M.A., B.Sc.; J. L. Rutgers Morgan, M.A., Ph.D.; Edgar Ford Morris, M.A.; Herbert Newall Morris; Ernest Brooks Naylor, B.Sc.; James Fraser Readman; Walter A. Riley; James McConnell Sanders; Fred. Pilkington Sargeant; Edward Shrapnell Smith; James Hart-Smith; Bertram Vincent Storr, B.Sc.; Archie Hugh Strong; Gustavus A. Waterhouse, B.Sc.; Leonard Philip Wilson.

Messrs. A. W. Crossley, F. J. M. Page, and E. W. Voelcker were appointed to audit the Society's accounts.

Of the following papers, those marked * were read:—

*17. "*Ammonium Amidosulphite.*" By Edward Divers and Masataka Ogawa.

When dry enough, ammonia does not combine with sulphur dioxide at a low temperature. When less dry, it does so, with such rise of temperature that the product of the union is at once partly decomposed. But by dissolving the ammonia in dry, alcohol-free ether, and passing the sulphur dioxide into the solution, kept very cold, the product is precipitated undecomposed. It is colourless, very deliquescent, and unstable in the air. It dissolves in water with a hissing sound, and, when the water has been well cooled, yields a solution which gives the reactions of ammonium sulphite only. It is also very soluble in absolute alcohol, forming the salt lately described by the authors, ethyl ammoniumsulphite. It is formed from 2 mols. ammonia and 1 mol. sulphur dioxide. In a dry atmosphere, at temperatures such as 35°, it loses nearly half of its ammonia, as well as a little water. After the decomposition, no sulphite-forming substance remains, and the mass is somewhat orange-coloured from the presence of a very small quantity of a red substance. A very little sulphate is also present, but the residue consists mainly of substances the composition and nature of which are not yet fully determined. They seem to be thio-amido-sulphonic compounds, as yet unknown, which, by heat, by spontaneous partial hydration, or by treatment with ammonia in alcoholic solution, yield sulphur, sulphur dioxide, sulphate, thiosulphate, amidosulphate, imidosulphate, and, almost certainly, sulphamide; when dissolved in water they give a reaction with silver nitrate, closely resembling, yet not that of a trithionate, for when acidified, their solution gives no sulphur or sulphur dioxide, or only mere traces, until it has been heated under pressure or for a long time.

The undecomposed salt appears to be correctly named *ammonium amidosulphite*, and to have the formula $NH_2 \cdot SO_2 \cdot NH_4$. The preparation of this salt shows Rose's assertion, that the gases unite only in equal volumes forming an orange substance which at once decomposes with water into sulphate and trithionate, to be altogether wrong and to be due to his having failed to prevent the decomposition of the product first formed.

*18. "*On the Products obtained by Heating Ammonium Sulphites, Thiosulphate, and Trithionate.*" By Edward Divers and Masataka Ogawa.

The authors have ascertained that *anhydrous ammonium sulphite* and *pyrosulphite* sublime unchanged at about 150° in a current of dry nitrogen, the vapours being no doubt the dissociated gases. At a temperature somewhat higher, *ammonium trithionate* decomposes into sulphur dioxide, sulphur, and ammonium sulphate. At about 150°, *ammonium thiosulphate* decomposes almost entirely, leaving a residue of sulphur and giving a sublimate of anhydrous ammonium sulphite; very small quantities of ammonia and hydrogen sulphide are also given off, giving rise possibly to a trace of trithionate. According to Spring, ammonium thiosulphate sublimes unchanged, except for temporary dissociation. To obtain the above results, moisture must be absent: the effect of slowly heating *hydrated ammonium sulphite* in a current of dry nitrogen is partly to dehydrate it, partly to convert it into pyrosulphite, at about 120°; and then at about 150°, the residue decomposes into sulphur dioxide and ammonium sulphite (approximately hemihydrated) which sublimes.

Ammonium trithionate, a very deliquescent and unstable salt, not hitherto described, was prepared from the potassium salt by the use of hydrofluosilicic acid and ammonia, being much too soluble to admit of preparation by Plessy's process for the potassium salt. *Ammonium sulphite* was conveniently precipitated by saturating its concentrated solution with ammonia in a cooling mixture, and the *pyrosulphite* by similarly saturating its solution with sulphur dioxide. *Anhydrous ammonium sulphite* was obtained by exposing the hydrated salt in a desiccator over potash in an ammoniacal atmosphere.

*19. "*The Colour of Alkali Nitrites.*" By Edward Divers.

As Boguski (*J. Russ. Chem. Soc.*, 1899, xxxi., 543) has published his observations that a solution of sodium nitrite had a slight yellow colour which he could not remove, and which he attributes to the presence of an impurity, the author wishes to recall attention to his own statement (*Trans.*, 1899, lxxv., 86) that alkali nitrites have a slight yellow colour, especially in solution, and to Groves's statement in denial of its accuracy (*Proc.*, 1898, xiv., 222). The author desires now to claim accuracy for his own statement. The colour of the potassium salt is much more manifest than that of the sodium salt, principally because of its ability to yield exceedingly concentrated solutions. Solutions of potassium nitrite three parts of salt in one of water, are strongly yellow, particularly when thoroughly free from turbidity. Solutions of equal strength are equally coloured, whatever has been the source of the nitrite. Alkali nitrites are yellow in the fused state. The author is confident that Boguski's promised endeavour to remove the colour of sodium nitrite will be as futile as were those of chemists, long years ago, to remove the pink colour from manganous salts. Boguski's observation that the solid nitrite is sometimes apparently without colour, depends upon the well known optical effect of fine division in lessening the colour of transparent solids.

"20. "*Solubility of Mixed Potassium Nitrite and Nitrate.*" By Edward Divers.

By crystallising out, as far as possible, the nitrate which is mixed with potassium nitrite in its usual method of preparation, there remains a mixture of about three parts of nitrite to one part of nitrate, and this constitutes the ordinary nitrite of commerce. This mixture is soluble in one-fourth its weight of water, although the pure nitrite requires one-third of its weight, and the pure nitrate about four times its weight of water to dissolve it. Accordingly, when a solution of three parts pure potassium nitrite in one part water is digested with powdered potassium nitrate at the common temperature, it readily dissolves up one part of this salt.

Discussion.

In reply to remarks by Messrs. Baker, W. P. Bloxam, and Scott, Dr. Divers said that the ammonium amidosulphite rapidly deliquesced in air, and he quite agreed with Mr. Bloxam's observation as to the great activity of hydrated ammonium sulphite with regard to atmospheric oxygen. As Rose had taken no precautions to maintain a low temperature during the reaction, there was no doubt that the compound resulting from the combination of two volumes of ammonia with one of sulphur dioxide gave off half of its ammonia again under the influence of the high temperature produced in the reaction.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, February 23rd, 1900.

Prof. S. P. Thompson, F.R.S., Foreign Secretary, in the Chair.

Prof. R. W. Wood, of the University of Wisconsin, U.S.A., exhibited and described :—

1. *Photographs of Sound Waves and the Kinematographical Demonstration of the Evolutions of Reflected Wave fronts.* The sounds were produced by electric sparks and photographed by means of the light emitted by carefully timed subsequent sparks, according to the method described in the *Phil. Mag.* for last year. The photographs included :—

(*a*). The reflection of a spherical wave, as a spherical wave from a plane surface.

(*b*). The reflection by an ellipse of circular waves from one focus, and the concentration of the waves as circular waves at the other focus.

(*c*). The plane wave-front formed by the reflection of a spherical wave at a parabolic surface.

(*d*). The wave-front formed by a spherical wave incident on a spherical surface.

(*e*). The wave-front formed by a plane wave incident on a spherical surface.

In cases (*d*) and (*e*) the wave-fronts are complicated and contain cusps. Prof. Wood pointed out that the paths of the cusps on the wave-fronts traced out the caustic curves.

In the following cases the wave-fronts were drawn for one hundred successive positions, and the evolution of the reflected wave was made clear by means of a kinematograph :—

(*a*). Plane wave on a hemispherical mirror.

(*b*). Spherical wave on a hemispherical mirror, and—

(*c*). Circular wave inside a complete circular mirror.

2. *A New Seudoscope.*—In this instrument the real and inverted images formed by two convex lenses are viewed stereoscopically. The inversion of the object viewed causes the relief to be reversed.

3. *Diffraction Colour-photographs.*—Prof. Wood showed some coloured photographs taken by his diffraction process. The principle of the method is based upon the tri-colour theory. Different colours are produced by gratings so ruled and arranged as to throw upon the eye the particular constituents of the required colours. The arrangement of gratings necessary to produce a coloured picture is obtained by photographing properly spaced gratings through red, green, and blue chromograms of the object. The superposition of one grating upon another, which occurs in this process, gives rise to an in-and-out-of-step arrangement, which produces secondary spectra. These, however, seldom affect the picture to any serious extent.

4. *Artificial Parhelia.*—When printing fine gratings upon gelatin, if the film is too thick, no print is formed, but the gelatin warps. If such a film is placed in a converging beam, the central image is accompanied by four marked concentrations of light situated at the extremities of two diameters at right angles. An examination of one of these plates with a microscope shows that there is a ridge for every third line of the grating, and that the plate is crossed at right angles to these lines by irregularly spaced cross ridges.

Prof. Wood also exhibited some photographs taken by zone plates, a silvered copy of a Rowland grating, a photograph of a dynamite explosion, the motion of a ball in its flight, and the anomalous dispersion produced by a cyanine prism.

Mr. Boys gave some details concerning the photograph of the explosion shown.

Prof. Everett expressed his interest in the demonstrations.

Prof. Herschel asked if the photographs of sound waves after reflection had been verified by comparison with waves on mercury.

Mr. Watson pointed out that this could not be done, as it is impossible to get a solitary wave on the surface of mercury. Owing to the dependence of velocity on wave-length, any such solitary wave draws out into a train of waves.

The Chairman proposed a vote of thanks to Prof. Wood, and announced that, by invitation of Prof. Callendar, a Special Meeting will be held in University College on March 2nd. The Meeting then adjourned.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

Ordinary Meeting, February 20th, 1900.

Prof. Horace Lamb, M.A., F.R.S., President, in the Chair.

"*Some Criticisms on the Modern Theory of Solutions.*" By Edgar F. Morris, M.A.

By applying the ordinary assumptions of the kinetic theory of solutions to the case of a semipermeable cell depressed below the surface of the solvent, the result is deduced that the percentage composition of any solution is a linear function of its density. The form of the reaction equation for the catalysis of esters shows that the action cannot be attributed to independently moving ions. Other facts disproving this theory are the occurrence of electrolytic solutions with normal molecular weights, and of cases where the molecules would have to be regarded as split into most curious fragments to provide a sufficient number of ions—in the case of certain metals in mercury solution into more ions than atoms. Prof. Fitzgerald has previously shown the physical basis of this theory to be unsound, and, as the chemical applications give untrue results, it would appear to be time to abandon the theory.

Borates of the Magnesium Series.—L. Ouvrard.—The author's experiments lead him to believe that the methods used (by the wet way) for preparing definite tribasic borates are not satisfactory, owing to the easy decomposition of the borates by water. If, however, the same borates are prepared by the dry method, which the author describes, the body prepared presents in general a great amount of stability towards various agents of decomposition.—*Comptes Rendus*, cxxx., No. 6.

CORRESPONDENCE.

LAW OF ATOMIC WEIGHTS.

To the Editor of the Chemical News.

Sir,—If the process of arranging the elements according to the law discovered by Prof. Delaunay, and described in a letter to the Chemical News (vol. lxxxi., p. 70), is continued, the addition of the atomic weights of the several pairs of elements gives the series:—

Na=23·05, K 39·11, Cu 63·6, Rb=85·43, Ag=107·92.

A somewhat curious relation is to be noticed between the atomic weights of these elements, for by adding the atomic weight of sodium to that of any other in the series a number is obtained which approximates to the atomic weight of the next element in the series, thus:—

Na + K = 23·05 + 39·11 = 62·16 = Cu − 1·44
Na + Cu = 23·05 + 63·60 = 86·65 = Rb + 1·22
Na + Rb = 23·05 + 85·43 = 108·48 = Ag + 0·56

I should like to point out an error which occurs in Prof. Delaunay's third list; Cr is left out, and should occupy the place of V, and V the place of Ti (in brackets) in his list.—I am, &c.,

Nemo.

NOTICES OF BOOKS.

The Chemistry of Vegetation and Agriculture. ("Chimie Végétale et Agricole"). By M. Berthelot, Senator, Permanent Secretary of the Academy of Science, &c. In Four Volumes. Paris: Masson et Cie. 1899. Pp. 1997.

Synthetical chemistry, which has occupied M. Berthelot's attention to such a great extent since the year 1853, effects in the laboratory what plant-life does in the field. The author effected the synthesis of the carbides of hydrogen and the alcohols, by starting from carbonic oxide, carbonic acid, and water,—that is to say, the simple mineral compounds which are acted on by vegetable organisms.

In 1860, when the author published the general methods by which he had attacked and overcome the problem of purely chemical synthesis, he also pointed out the importance of biological synthesis,—that is to say, synthesis carried on under conditions compatible with the natural phenomena of life. The realisation of these ideas was reached when a portion of the estate of the Chateau of Meudon was placed at his disposal in 1883. The results obtained at this experimental station have been published year by year, and the details of the experiments and the analyses made have been given in the *Annales de Physique et de Chemie*. The time has now come to collect all these scattered notes and papers in one publication, setting forth the whole co-ordinated system.

The fundamental principle of these experiments has turned on the study of the fixation of the free nitrogen of the atmosphere by the soil and by vegetation, under the influence of the micro-organisms present, and under the action of the silent atmospheric electricity.

The discoveries made by the author in this respect have wrought a great change in the ideas held by the best authorities on the negative *rôle* of the free atmospheric nitrogen in vegetation. Thirty years ago free nitrogen was not considered to have any influence on vegetation or agriculture, but this opinion has since been entirely altered. He shows, by experiments spreading over seven years on sterile soil (that is, deprived of microbes), such as argillaceous sand, and then true vegetable mould, that certain of these sterile soils have the power of fixing nitrogen, as well in the absence of higher vegetable life as with its help. The influence of leguminoses on the fixation of nitrogen, so long affirmed by M. George Ville, though not then accepted, has been confirmed and explained by M. Berthelot's discoveries with regard to the microbes in the soil and their action in the mechanism of the assimilation of nitrogen. The sum of these various discoveries constitutes a new doctrine, but there is also a third cause of the fixation of nitrogen, and this is a purely chemical one.

These are the questions which are dealt with in vol. I.

The second volume contains the author's researches on the general progress of vegetation, which gives a sort of ponderable chemical equation of an annual plant during the whole of its evolution, from its first sowing up to the time when it reproduces the seed at the end of its development: this subject has not previously been dealt with systematically, in spite of its importance.

Volume III. is on special researches on vegetation and its products; in it the author examines the presence and distribution of certain special elements in plants, such as sulphur, phosphorus, silica, &c.; the formation of oxalic acid and carbonates; the formation and partition of nitrates in vegetation; and the emission of carbonic acid and absorption of oxygen by the detached leaves of plants. He then treats of the decomposition of sugars by purely chemical means, with the production of carbonic acid, &c. A special part of this volume consists of the experiments and observations made at various times on the definite principles having the double power of combining with free oxygen and then giving it up to bodies which oxygen will not oxidise directly—a phenomenon connected with both oxidising ferments and the microbial fixation of nitrogen.

Volume IV. contains two distinct parts, the one general, relating to soils, their composition, &c., and the other relating specially to wine, and the influence of soils on its bouquet.

It is not only the chemical and biological side of the question that is dealt with in so masterly a manner in this work, but also the practical side by which these phenomena can best be adapted and applied to agriculture and its allied industries, to the advantage and profit of mankind.

This is a wide and important subject, and will demand very serious attention before many years have passed over our heads.

First-stage Magnetism and Electricity: treated from the Stand-point of Potential and Potential-gradient. By R. H. Jude, M.A., D.Sc. With 138 Illustrations. London: W. B. Clive, University Correspondence College Press. Pp. 350.

It has often been noticed by teachers, as well as by examiners, that one of the greatest difficulties met with by students is the conception of "Electrical Potential." The difficulty lies not in the mathematical aspects of the subject, but in attempting to apply the idea of potential to the general explanation of electrical phenomena. In the opinion of the author the trouble arises from two causes; viz., that the idea of potential is not brought before the student until too late, and from the fact that man has no "electrical sense," such as he has of heat, sound, light, &c. The object of this book is to overcome both these difficulties. The notion of potential is brought before the student very early in the course, so that he may the more easily assimilate and become accustomed to it. This subject, and electrostatics in general, form Part I. of this volume; in Parts II. and III. the aim of the author has been to set forth the fundamental principles of electrodynamics and magnetism as clearly as possible: in this he has succeeded, and has given us a book which will no doubt be fully appreciated by beginners in electrical science. A number of exercises and examination papers are included in the volume, but there is one point which

might be improved: the author is a great deal too fond of italics; in fact, there is not a page on which this form of emphasis is not used several times.

Richter's Organic Chemistry, or Chemistry of the Carbon Compounds. Edited by Prof. R. Anschütz. Authorised translation by Edgar F. Smith, Professor of Chemistry, University of Pennsylvania. Third American, from the Eighth German Edition. Vol. I. *On the Aliphatic Series.* With illustrations. London: Kegan Paul, Trench, Trübner, and Co., Lim. 1899. Pp. 625.

As the second edition was a great improvement on the first, so does the third show a great advance beyond the second. Indeed, the book is very different from the earlier editions, and has undergone many radical changes.

The marvellous advances in the various lines of synthetic organic chemistry have made it necessary to effect considerable changes in the text, and it has been found better for practical reasons to issue this new edition in two volumes.

Volume I. "On the Chemistry of the Aliphatic Series," is divided into eight parts, beginning with the hydrocarbons, their halogen derivatives, the oxygen derivatives of the methane hydrocarbons, and successively, the mono-, di-, tri-, tetra-, penta-, hexa-, and polyhydric alcohols and their oxidation products.

The work is well and clearly printed and provided with an excellent index of 36 pages of two columns each.

Volume II. is in rapid preparation, and will be published shortly.

Handbook of Chemical Analysis. ("Manuel d'Analyse Chimique"). Applied to the Examination of Industrial and Commercial Products. By Emile Fleurent. Paris: G. Carré and C. Naud. 1898. Pp. 582.

This work does not pretend to be a complete treatise on chemical analysis, but a practical handbook, destined to serve as a guide to those engaged with commercial chemical products. The author has endeavoured to realise three special objects; to describe, without overburdening them with theoretical details, the general methods of qualitative and quantitative inorganic analysis, and elementary organic analysis; to simplify the examination of materials and to save time, by only giving one or two methods by which the results can be rapidly obtained; and to collect together in one volume the methods of examination of the most important, as well as the most diverse products, both metalloids and metals, chemical and organic manures, vegetable and animal products, fermented drinks, &c., &c.

Numerous tables are given at the end of each chapter giving in a practical form the results obtained by applying the methods described in the text.

Outlines of Theoretical Chemistry. By Lothar Meyer, Professor of Chemistry in the University of Tübingen. Translated by P. P. Bedson, M.A. (Dun.), D.Sc. (Lond. and Dun.), F.C.S., &c.; and W. C. Williams, B.Sc., F.C.S., &c. Second Edition. London, New York, and Bombay: Longmans, Green, and Co. 1899. Pp. 240.

In this work the author has abstained from giving numerical results of observations and measurement, or detailed descriptions of experimental methods, but has confined himself as far as possible to theoretical speculations which, though of value to the student, are also of interest to those who, not wishing to be troubled with details of investigations, are anxious to become familiar with the general conclusions arrived at. Though retaining the original form of the first edition, this, the second edition, has been brought up-to-date, by footnotes and appendices.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 6, February 5, 1900.

Superficial Tension of some Organic Liquids.—Paul Dutoit and Louis Friderich.—The authors have measured the temperature coefficient of superficial molecular energy of a group of organic liquids. This research verified the relations found by Ramsay and Shields between the coefficient of temperature and the molecular weight of the liquid. The authors find that—(1) The coefficient of temperature varies with the temperature for liquids which do not retain the same molecular size during the interval of temperature under consideration; (2) this coefficient is independent of temperature for normal liquids; (3) for normal liquids this coefficient does not represent a single constant for all bodies, but varies between certain indefinite limits, which have not yet been determined. With regard to the two first points, the authors' results confirm those published by Ramsay and Shields.

Volumetric Estimation of Hydrogen and Chemical Tensions.—Alb. Colson.—Oxide of silver has the power of absorbing hydrogen without acting either on the saturated carbides or on oxygen. M. Fouqué, in his researches on volcanic gases, separated the hydrocarbides from hydrogen by shaking up the gas with alcohol and dissolving the carbides. The author proposes an inverse method; since this latter is independent of the coefficient of solubility. Oxide of silver behaves as if it emitted vapours in hydrogen at all temperatures, and as if hydrogen acted principally on these vapours. The author's experiments show the mechanism of diffusion of solids in gases.

Action of Concentrated Ammonia on Mercurdiammonium Iodide.—Maurice François.—Dimercurammonium iodide, otherwise called tetramercurammonium iodide, is produced when mercuric iodide is heated with a large excess of concentrated ammonia. The reaction is represented by the equation $2HgI_2+4NH_3+H_2O=3NH_4I+Hg_2NIH_2O$. The dimercurammonium iodide is insoluble, and remains suspended in the solution of ammonium iodide. By studying this reaction, the author finds that it is limited and reversible. At the moment of equilibrium, a given volume of the ammoniacal liquor contains a quantity of free ammonium iodide, which is constant for a given temperature and for a given concentration of ammonia.

Acidimetric Value of Substituted Malonic Acids compared with those of corresponding Normal Diacids.—G. Massol.—The author determines numbers under identical experimental conditions with those under which similar numbers of the diacids were determined; he compares them, and draws the following conclusions:—(1) For normal acids of the oxalic series increase of molecular weight leads at first to a progressive diminution of the acidimetric value, corresponding to about 2 cal., then to 1 cal., for each CH_2 added. (2) For the homologous series of mono-substituted malonic acids the phenomenon is identical; the quantity of heat evolved decreases at first with increase of molecular weight, then remains constant. In this case, however, the minimum is sensibly higher than in the former series. (3) If each mono-substituted acid is compared with its normal homologue containing the same number of atoms of carbon—and having, consequently, the same molecular weight—it is found that the abnormal acid evolves more heat than the normal. (4) These results confirm the conclusion, which the author has already announced, that the acidimetric value of polyacids depends much more on their molecular structure and

the separation of the carboxyls than on their molecular weight.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxi., No. 22.

Properties of Calcium.—Henri Moissan.—Already inserted.

Preparation of Lithium-ammonium, Calcium-ammonium, and of the Amidides of Calcium and Lithium.—Henri Moissan.—When liquid ammonia cooled to −50° is brought into contact with calcium or lithium an intensely blue coloured solution is produced. The methodical study of the properties of calcium led the author to examine the reactions produced by calcium in the presence of gaseous and liquid ammonia. Wishing to find the temperature limit at which the alkaline ammoniums could be formed, he experimented with the four metals potassium, sodium, lithium, and calcium. Each metal was placed in a U-tube connected with a supply of dry ammonia gas; these were placed in a water-bath; for low temperatures, the water was replaced by acetone and the temperature diminished by adding small pieces of solid carbonic acid; in this way the temperature could be brought down to −75°, and the ammonia gas solidified. In these experiments the ordinary atmospheric pressure was always maintained. The following results were obtained:—

Lithium is attacked at			+70°	with liquefaction.
Calcium	,,	,,	+20°	without ,,
Potassium	,,	,,	−2°	with ,,
Sodium	,,	,,	−20°	with ,,

These figures also represent the temperatures of decomposition of the ammonium metals at the ordinary pressure in a current of ammonia gas. To establish the composition of lithium-ammonium, two methods were followed. Experiments I. and II. were made by starting with a known weight of the metal, and weighing the ammonia combining with it; Nos. III. and IV. were obtained by preparing lithium-ammonium as described above, and estimating the ammonia by Schlœsing's method.

	I.	II.	III.	IV.	Theory for NH_3Li.
Lithium ..	28·07	28·40	28·72	28·82	29·16
Ammonium ..	71·93	71·60	71·28	71·18	70·83

The composition of calcium-ammonium was found by similar means; Nos. I., II., and III. by synthesis, and No. IV. by estimating the calcium and the ammonia in a known weight of the compound.

	I.	II.	III.	IV.	Theory for $(NH_3)_4Ca$.
Calcium ..	37·04	37·60	37·41	37·44	37·03
Ammonium ..	62·96	62·40	62·59	62·51	62·96

Action of Acetylene on the Ammonium Metals.—Henri Moissan.—When we cause the alkaline metals to react on the different carbides of hydrogen and on organic compounds, the decomposition is often very violent. As soon as the temperature rises, complicated phenomena of polymerisation and hydrogenation intervene, and soon hide the principal reaction, but the facility with which the ammonium metals form compounds at a low temperature gives us a new method with a much slower reaction. After fully describing the apparatus and the manipulatory details, the author describes the action of acetylene, of sodammonium, potassammonium, lithium-ammonium, and calcium-ammonium, by which he obtained the following acetylenic acetylides:—$C_2K_2C_2H_2$, $C_2Na_2C_2H_2$, $C_2Li_2C_2H_2.2NH_3$, $C_2Ca_2C_2H_2.4NH_3$.

Preparation and Properties of an Organic Ammonium: Lithium-monomethylammonium.—Henri Moissan.—When methylamine is passed over metallic lithium at −20°, it condenses and wets the metal; abundant blue striæ are formed, and the temperature rises; the metal soon disappears, leaving a blue liquid. When the solution is complete, an excess of methylamine is allowed to condense, and the current of gas then cut off, the tube is taken out of the refrigerator, and allowed to take the temperature of the laboratory. The product is then very thick and of a dark blue colour, approaching black; its composition is very close to the formula $N\begin{cases}CH_3\\H\\H\\Li\end{cases}+2N\begin{cases}CH_3\\H,\\H\end{cases}$ which corresponds to $(NH_2CH_3)_3Li$.

Colour of Carbide of Calcium.—Henri Moissan.—Already inserted (see p. 67).

The Preparation and Properties of Crystallised Phosphide of Calcium.—Henri Moissan.—Already inserted (see p. 87).

Heat of Formation of Quicklime from its Elements.—Henri Moissan.—Already inserted (see p. 89).

Preparation of the Arsenides of Strontium, Barium, and Lithium in the Electric Furnace.—P. Lebeau.—Already noticed in this column (vol. lxxx., p. 59).

Speed and Limit of Etherification of Phosphoric Acid by Glycerin.—H. Imbert and G. Belugou.—The return of glycerophosphoric acid when we act with more or less hydrated phosphoric acid on glycerin at various temperatures, depends principally, not only on the state of hydration of the acid and the temperature, but also on the time.

Salicylic and Paraoxybenzoic Aldehydes and Salicylhydramine.—M. Delépine and P. Rivals.—Already noticed (vol. lxxx., p. 295).

Metallic Derivatives of Salicylhydramide.—M. Delépine.—The author is of the opinion that the most logical way of expressing the constitution of the salicylhydramides is by making them derivatives of the salicylimides, of which Schiff has described the cupric compounds of the type $Cu<^{O-C_6H_4-CH=N-R}_{O-C_6H_4-CH=N-R}$. Methylsalicylimide of copper is obtained by adding an alcoholic solution of acetate of copper in methylamine to an alcoholic solution of salicylic aldehyde. An emerald-green precipitate is formed, crystallising in needles; re-crystallisation in alcohol enables the methylsalicylimide to be obtained in dark green, almost black, crystals, several c.m. long. On analysis it gave 19·29 per cent of Cu.

Heat of Oxidation of Tungsten.—M. Delépine and L. A. Hallopeau.—Already inserted (see p. 66).

On the Symmetric Dinitrodixylyl- and the Dinaphthyl-Carbamides.—H. Vittenet.—Two molecules of nitro-5-oxylidine-1:3:4 and one molecule of $COCl_2$ in toluene at 20 per cent was heated for an hour in a sealed tube at 130°. A brown mass was obtained, which was washed with cold benzene, then warm water; it was then crystallised in acetic acid. Analysis gave N = 15·58. Another body, containing N = 15·74, was obtained in a similar manner by heating two molecules of nitro-6-xylidine-1:3:4 in the same way.

On Chloracetate of Phenyl and its Reactions.—A. Morel.—The author describes the preparation of chloracetic ethers, and the action of alcohols and of ammonia on chloracetate of phenyl. NH_3 reacts on the phenolic ether function to give an amide before reacting on the alcoholic chloride function to give an amine; it was also noticed that alcohol very rapidly transforms $\begin{matrix}CH_2Cl\\|\\COOC_6H_5\end{matrix}$ into $\begin{matrix}CH_2Cl\\|\\COOC_2H_5\end{matrix}$ in a sealed tube at 130°.

Reactions of Chloracetate of Phenyl and Glycolate of Phenyl.—A. Morel.—The comparison between the reactions of the monochlorated chloride of acetyl, of chloracetate of benzenol, and of the double glycolate of benzenol on the one hand, and of oxychloride of carbon, chlorocarbonate and neutral carbonate of benzenol on the other hand, show that these two groups belong to

two different classes. The first are clearly derived from a body of complex functions, the second from a symmetric body.

Cyclic Isomerisation of Methyloctanedienonol.—G. Leser. — The product of condensation of methylheptenone with formic ether may be regarded as a body with an aldehydic function, methyloctenonal, $CH_3>C=CH-CH_2-CH_2-CO-CH_2-CHO$; or, as its tautomer, the secondary incomplete alcohol, methyloctadienonol, $CH_3>C=CH-CH_2-CH_2-CO-CH=CH-OH$. The author added 60 grms. of the latter to 300 grms. of sulphuric acid at 80 per cent, and agitated until dissolved. There was no sensible heating, but only a brown colouration of the liquid. The whole was boiled for a few minutes on the water-bath, then cooled and diluted with water; the citron odour has disappeared and given place to a faint but agreeable smell. The aqueous solution is extracted with ether; the ethereal layer, washed with water, is distilled. At 115—117°, under 17 m.m., a liquid is isolated; analysis shows it to be an isomer of methyloctadienonol, $C_9H_{14}O_2$.

On the Aminocampholenes.—E. Blaise and G. Blanc.—Already noticed (see vol. lxxx., p. 72),

Apparatus for the Estimation of Carbonic Acid in Mineral Waters.—A. Held.—This paper requires the accompanying diagram.

MISCELLANEOUS

On Parapyruvic Acid.—L. Wolff.—The pyruvates in aqueous solution slowly undergo a modification and become transformed into the so-called syrupy salts of pyruvic acid. This was known by Berzelius and Voelckel. This modification is much more rapid in the presence of traces of certain condensation agents (CNK, KOH, NH_3), and leads to the transformation of neutral salts of a bibasic acid, $C_6H_8O_6$. This acid, which may be called pseudopyruvic, results from the condensation of 2 molecules of pyruvic acid; but in the free state it partly splits up into water and lactonic acid, $C_6H_6O_5$ (keto-valerolactonecarbonic)—

$$2CH_3.CO.CO_2H = CH_3.C(OH)(CO_2H)-CH_2-CO-CO_2H = CH_3-C(CO_2H)(O-CO)-CH_2-CO + H_2O.$$

As for the free pyruvic acid, it also undergoes a transformation. Whether the modified acid contains the same mixture has not yet been proved, but a crystallisable acid, $C_6H_6O_5$, is produced, fusible at 116—117°, which still requires examination; it is not pyrotartaric acid. *Neutral Parapyruvates.*—The barium salt, $C_6H_6O_6Ba+3\frac{1}{2}H_2O$, is obtained by neutralising CO_3Ba by means of a solution of pyruvic acid (distilled at 63—66° under 30 m.m.), then adding a little CNK or KOH. A granular mass is formed, becoming anhydrous at 115°; it is then very hygroscopic. It is slightly soluble in water, easily so in acetate of soda and hydrochloric acid. Digested with acetic acid it takes a chalky appearance, and in that state contains $4\frac{1}{2}H_2O$ ($3\frac{1}{2}H_2O$ is given off in vacuo, the remainder at 120°). The calcium salt ($+4H_2O$), is similar to the barium salt, but more soluble. The lead salt is obtained by the addition of acetate of lead to a solution of pyruvic acid; the pyruvate of lead is not known, the acetate acting as a condensation agent. The salt of zinc is obtained in the same manner in the form of small plates or a granular powder. The acid set at liberty from the barium salt is a colourless syrup which, according to analysis, is a mixture of 20 per cent of parapyruvic acid, and 80 per cent of lactonic acid, $C_6H_6O_5$. When we endeavour to transform parapyruvic acid entirely into its lactone by heating to 70°, it is split up into CO_2 and methyldihydrotrimesic acid. Concentrated soda produces the same reaction. Neutralised by alkalis the mixture of acid gives a parapyruvate; with carbonates in the cold we obtain lactonates; it is in this manner that the salt of barium is obtained $(C_6H_5O_5)_2Ba$, which is very soluble and precipitated by alcohol in colourless flakes; its aqueous solution is slightly acid, but gradually, especially when warmed, the parapyruvate is deposited, and the solution becomes very acid. The acid set at liberty from the lead salt by SH_2 is distinguished from that formed by the barium salt, in that it is transformed into a crystalline compound which gives phenylhydrazone fusible at 200°.—*Liebig's Annalen. Chem.*, cccv., p. 154.

On the Constitution of Tartarazine.—R. Anschutz.—Tartarazine is split up by reduction into soda salts of sulphanilic acid, and aminotartarazinogenic acid, $C_{10}H_8N_3SO_3H$:—

$$CO_2Na-C=N-C=\!=NHC_6H_5(SO_3Na)_4 + H_4 = CO-N.C_6H_4(SO_3Na)_4$$

$$= NH_2C_6H_4SO_3Na + CO_2Na-C=N-CH-\!-NH_2 \quad (CO\,.\,N\,.\,C_6H_4\,.\,(SO_3Na)_4)$$

Monosodic aminotartarazinogenate, $C_{10}H_8N_3SO_3Na$, is obtained by heating a solution of 80 grms. of tartarazine and 40 grms. of NaCl in 800 c.c. of water, with 100 grms. of powdered zinc, until decolouration occurs, and then pouring the solution, which takes a violet colour when exposed to air, into 300 c.c. of concentrated HCl. Colourless needles, which are a mixture of the two acids, are precipitated. This mixture, weighing 30 grms. is dissolved in 200 c.c. of water, with the addition of 10·6 grms. of dry CO_3Na_2. On the addition of 5 c.c. of HCl at 33 per cent, the soda salt in question, is precipitated in small colourless crystals. The free acid, $C_{10}H_8N_3SO_3H$ (sulphoxyphenyl-4-amidopyrazolone-3-carbonic acid), crystallises in dilute HCl in small slightly coloured heavy needles, almost insoluble in water, alcohol, and acetic acid, and slightly soluble in warm aqueous alcohol and warm HCl. Its solution in alkaline carbonates takes a violet colour on contact with the air. When heated it decomposes without melting.—*Liebig's Ann. Chem.*, cccvi., p. 1.

The Action of Semi-carbazide on Oxide of Mesityl.—C. Harries and F. Kaiser.—Two semi-carbazones from oxide of mesityl are described; the one prepared by the usual methods melts at 156°; submitted to distillation it changes into its isomer, fusible at 129°. The first only is split up by acids into semicarbazide and oxide of mesityl. The second is obtained by leaving equimolecular aqueous solutions of chlorhydrate of semicarbazide and oxide of mesityl in contact, in the cold, for twelve hours. The result of the reaction is a solid basic body, fusible at 130—131°, and apparently having the formula of a pyrazolic derivative, and not that of a semi-carbazone. The corresponding picrate crystallises in long yellow needles, fusible at 136—137°, slightly soluble in cold organic solvents.—*Berichte*, xxxii., p. 1338.

Preparation of Adipic Acid.—O. Aschan.—Adipic acid has already been prepared by the oxidation of a carbide boiling at about 80°, obtained from petroleum from Baku. The author uses this reaction as the basis of the preparation of adipic acid. That portion of the petroleum ether boiling at 78—82° (d=0·752 at 15°) is utilised; it is heated in a retort fitted with a reflux condenser, with 10 parts of NO_3H(d=1·42). At the end of fifty or sixty hours, it is concentrated to one-quarter by distillation, then evaporated on a water-bath. The crystalline mass remaining is taken up with its own volume of cold water, and drained after twelve hours. It is then dissolved in NH_3 at 25 per cent, exhausted with ether, brought to boiling, and the acid precipitated by HCl. It is purified by crystallisation in boiling water, and fuses at 149·5°. 250 grms. of carbide give 44 grms. of adipic acid.—*Berichte*, xxxii., p. 1709.

MEETINGS FOR THE WEEK.

MONDAY, 5th.—Society of Arts, 8. (Cantor Lectures). "Photography of Colour," by E. Sanger Shepherd.
— Royal Institution, 5. General Monthly Meeting.
— Society of Chemical Industry, 8. "The presence of Naphthalene in Coal-gas," by R. W. Allen. "Notes on the Determination of the Iodine Value of Oils," by Arthur Marshall, A.I.C., F.C.S.
TUESDAY, 6th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
WEDNESDAY, 7th.—Society of Arts, 8. "Macombe's Country (South of the Zambesi), its Ancient Goldfields and Industrial Resources," by Dr. Carl Peters.
— Society of Public Analysts, 8. "The Determination of Carbon and Sulphur in Steel," by Bertram Blount, F.I.C. "Maize Oil," by Rowland Williams, F.I.C. "Note on the Assay of Creosote," by A. D. Hall, M.A. "Note on the Influence of Temperature and Concentration on the Saline Constituents of Boiler Waters," by Cecil H. Cribb, B.Sc., &c.
THURSDAY, 8th.—Royal Institution, 3. "Recent Excavations at the Argive Heræum (in Greece)," by Charles Waldstein, Litt.D., Ph.D., L.H.D.
FRIDAY, 9th.—Royal Institution, 9. "Bacteria and Sewage," by Frank Clowes, D.Sc., F.C.S.
SATURDAY, 10th.—Royal Institution, 3. "Polarised Light," by The Rt. Hon. Lord Rayleigh, M.A., D.C.L., &c.

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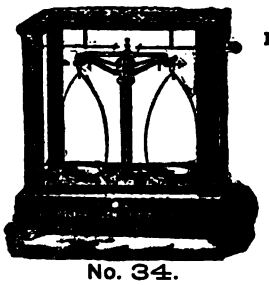

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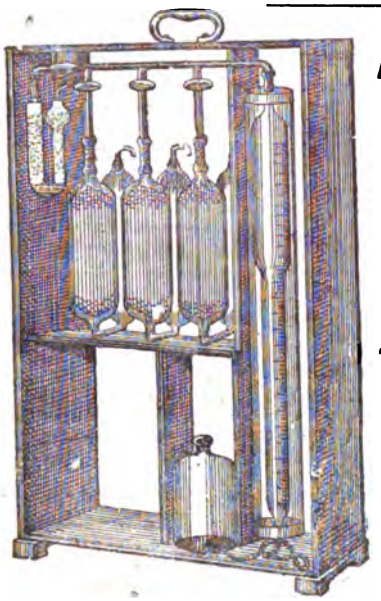

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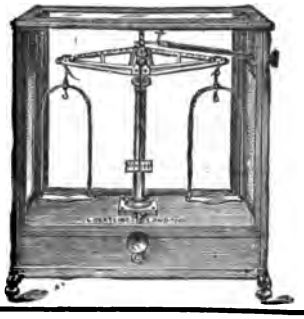

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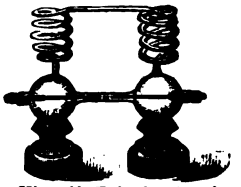

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THE CHEMICAL NEWS.

VOL. LXXXI., No. 2102.

THE ANALYSIS OF CAYENNE PEPPER.

By WILLIAM C. R. KYNASTON.

HAVING recently had occasion to examine some samples of Cayenne pepper, and not being able to find any recent work upon the subject, I have examined three samples of pepper of known purity with the following results:—

	A. Per cent.	B. Per cent.	C. Per cent.
Moisture	9·90	8·90	9·12
Ethereal extract	21·08	20·91	20·97
Alcoholic extract	9·54	10·43	15·12
Woody fibre	22·09	25·30	17·96
Total ash	6·27	6·50	5·66
Insoluble ash	1·22	1·51	1·10
Sand	0·11	0·06	0·16
Alkalinity of ash as K_2O	1·79	1·91	1·53
Percentage of alkalinity as K_2O in total ash	28·55	29·38	27·03

Sample marked A was ground in the laboratory from chillies.

Sample marked B was ground in the laboratory from Japan chillies.

Sample marked C was a sample of Cayenne pepper of known purity, as placed on the market.

The method of analysis was as follows:—

Moisture.—This item was the total loss at 100° C.

Ethereal Extract.—This was made by placing the dried sample in a dried filter-paper and extracting with ether, in a Soxhlet's apparatus, for a period of three hours.

Alcoholic Extract.—The residue from the ether extraction was completely extracted with hot alcohol.

Woody Fibre.—The residue from the alcoholic extraction was treated in the usual way for woody fibre.

The conclusions to be drawn from the above figures are:—

1. The ethereal extract seems to be practically constant.
2. The alkalinity of the ash seems to be practically constant.
3. The percentage of alkalinity to that of ash is fairly constant.

The Laboratory, 28, Chapel Street, Liverpool.

THE CHEMICAL EXAMINATION OF HOPS AND OF EXTRACTS OF HOPS.

By ERNST HANTKE.

THE analysis of hops and of extracts of hops comprises the following determinations:—The water, the matter soluble in petroleum ether, the wax, the matter soluble in sulphuric ether, the tannin, and the ash.

The estimation of the water is effected by drying the hops at 100—102° until the weight is constant; this requires eight hours. Extract of hops is intimately mixed with calcined sand and carefully heated to 100—102°; this operation requires about twenty-four hours, the whole mass becomes hard.

For estimating the matters soluble in petroleum ether, the hops are first kept at 50° for two days, after which they are further dried in the exsiccator for two days. Extract of hops is first mixed with calcined sand. The extraction is carried out in a Soxhlet extractor, and the petroleum ether used should boil at a temperature lower than 50°. Its density would then be from 0·6616 to 0·6620 at 15°. The extraction is complete after eight or nine hours. The petroleum ether is driven off on the water-bath and the residue dried. When it is necessary to estimate the soft resin in the matter soluble in petroleum ether, the residue is treated with alcohol at 90 per cent (by volume) at the ordinary temperature, shaking occasionally until complete solution is effected. This operation requires nearly a day. The colour of the solution gives certain indications as to the place of origin of the hops.

The alcoholic solution is filtered into a small tared flask, the residue well washed with alcohol, which latter is driven off by heat. The residue which consists of the pure soft resin is weighed.

The difference between the soft resin and the matter soluble in petroleum ether gives the wax.

The hard resin is determined by submitting the hops, already treated with petroleum ether, to the solvent action of anhydrous sulphuric ether (d=0·7283 at 15°). The extraction by ether proceeds fairly rapidly, and is generally complete after the lapse of three hours. The ether is then driven off and the residue weighed.

To estimate the tannin, 6 grms. of hops, or of extract, are boiled for five hours with 400 c.c. of water in a flask fitted with a vertical condenser; fill up to 500 c.c. and filter. 100 c.c. of the clear solution are taken and evaporated to dryness; this gives the soluble matter. The remainder of the filtrate is submitted to the action of a skin filter, and 100 c.c. of the solution containing the non-tanning material are evaporated down. The difference between the results obtained in the first place, and the residue obtained after the absorption of the tanning material enables us to estimate the quantity of tannin present.

The ash is determined in the usual manner.

The results of a large number of analyses shows that a pound of extract of hops is equal to 12 pounds of hops, a fact which is confirmed in practice.—*Chemiker Zeitung*, 1899, p. 545.

THE ESTIMATION OF BISULPHIDE OF CARBON,

By Dr. A. GOLDBERG.

THE method of estimating bisulphide of carbon which I am about to describe is based on the following theoretical considerations:—

(1). $CS_2 + 2NH_3 = CS{<}^{SNH_4}_{NH_2}$,

(2). $CS{<}^{SNH_4}_{NH_2} = H_2S + CNS.NH_4$,

(3). $CS{<}^{SNH_4}_{NH_2} + ZnO = ZnS + H_2O + CNS.NH_4$,

(4). $H_2S + ZnO = H_2O + ZnS$.

The different reactions take place in part successively, and in part simultaneously, but finally the quantity of zincic sulphide formed is equivalent to the quantity of sulphide of carbon which entered into the reaction, so that the calculation can be easily made according to the simple relation $CS_2 = H_2S = Zn$.

The reagents required are:—An ammoniacal solution of zinc of known strength, and a solution of sodic sulphide titrated by means of the zinc solution. The substance under examination is heated on a water-bath with alcoholic ammonia to a temperature of about 60°, but a temperature of 100° does no harm; the operation is carried out in a small flask stout enough to bear pressure, and the stopper should be firmly held in. Into this flask is poured 1 grm. of the sample of sulphide of carbon, 5 or 6 c.c. of ammonia (d=0·910), and about 25 c.c. of absolute alcohol;

it is then tightly closed and left to stand in a warm place. When the reaction is finished, the solution should be of a yellow colour; the contents of the flask are poured into a beaker and considerably diluted, a known volume of the ammoniacal solution of zinc is then added, and the whole heated to boiling with continuous stirring, and the excess of zinc determined by the aid of a titrated solution of sodic sulphide in the presence of nitro-prussiate of soda as an indicator.

When more than 0·2 grm. of bisulphide of carbon is taken for estimation, it is better to make up to a known volume, and then take an aliquot part for the titration.

The following are the results of a few estimations, made according to the method just described, on samples of perfectly pure bisulphide of carbon:—

Grm.		Grm.	Grm.	Per cent.
0·2178	CS_2 used	0·1861 Zn	= 0·2175 CS_2	= 91·86
0·4723	,, ,,	0·4041 ,,	= 0·4725 ,,	= 100·05
0·4502	,, ,,	0·2843 ,,	= 0·4494 ,,	= 99·82

It has been mentioned above that the solution has a yellow colour after heating; at the ordinary temperature, it is of a reddish yellow colour from the commencement. When diluted, after some hours, with a large quantity of water, it gives a perfect solution, but it absorbs a slightly smaller quantity of zinc. In this manner, a known quantity of bisulphide of carbon has given the following results:—

1. *Hot.*—(*a*) 0·2190 grm. Zn; (*b*) 0·2175 grm. Zn; (*c*) 0·2181 grm. Zn; (*d*) 0·2175 grm. Zn; (*e*) 0·2181 grm. Zn. Mean, 0·2180 grm. Zn = 0·2549 grm. CS_2 = 100·27 per cent.
2. *At the Ordinary Temperature.*—(*a*) 0·2123; (*b*) 0·2116; (*c*) 0 2116; (*d*) 0·2135; (*e*) 0·2116; (*f*) 0·2117 grm. Zn. Mean 0·2122 = 97·6 per cent of the theoretical amount.

This is probably due to the formation of a certain quantity of sulphocarbonate of ammonia at the ordinary temperature:—

$$CS\begin{matrix}<^{SNH_4}_{NH_2}\end{matrix} = H_2S + CNS.NH_4$$

$$CS_2 + H_2S = 2NH_3 + CS\begin{matrix}<^{SNH_4}_{SNH_4}\end{matrix},$$

a body which gives, during titration, $ZnCS_3$, which, on boiling, is mostly transformed into ZnS and CS_2. The result is that, with this secondary reaction taking place at the ordinary temperature, the relation is no longer $Zn = H_2S = CS_2$, but $Zn = 2H_2S = 2CS_2$.

The reaction is not changed if, instead of using alcoholic ammonia, ordinary aqueous ammonia be taken, but a longer time is necessary to complete the reaction. For example, 5 c.c. of bisulphite of carbon were treated with 30 c.c. of aqueous ammonia of 0·91 density, and heated for some days until the bisulphide of carbon had completely disappeared.

The solution, cooled to the surrounding temperature, was diluted to 250 c.c.; 10 c.c. required 0·2176 grm. of Zn (instead of 0·2174); that is, 100·09 per cent found.—*Zeitschrift für Angewandte Chemie*, 1899, p. 75.

The Preparation of Fuming Nitric Acid.—L. Vaning.—When we add concentrated nitric acid to formic aldehyde in solution, the liquid, in the cold, takes a yellow colour, and gives off abundant fumes of binoxide of nitrogen. This reaction can be utilised for the preparation of fuming nitric acid. In fact, if we react with polymerised formic aldehyde on nitric acid, fumes of binoxide of nitrogen are given off in the cold, but on moderately heating hyponitric acid is given off, which passed into nitric acid gives a product rich in binoxide of nitrogen. We can obtain an acid rich in nitrous fumes without distillation by gradually adding polymerised formic aldehyde.—*Berichte*, xxxii., p. 1392.

ON THE ABSORPTION OF IODINE BY PLANTS.

By P. BOURCET.

Different plants grown in the same soil absorb the various mineral substances contained by the soil in different proportions. Bunsen long ago observed that certain kinds of plants had the power of removing rubidium from soils in which this metal was present in such small quantities that it could not be detected even by spectrum analysis. Grandeau made similar observations with lithium and cæsium. It occurred to me that certain plants ought to absorb the iodine necessary to their structure and protoplasms from the soil or from water, and perhaps with the accompaniment of special functions. But as experiments on a large number of species would have taken me too far, I have limited these first experiments to a certain number of edible plants, my intention being to study at the same time how the iodine was introduced by alimentation into the animal economy.

Several cubic metres of earth were well broken up and made thoroughly homogeneous by several successive siftings. Five samples taken at hazard in the mass were found to contain an average of 0·83 m.grm. of iodine per 100 kilogrms. of soil. This earth was spread out, and a certain number of seeds were sown in it. The crops were gathered as they reached maturity, and the iodine they contained was estimated by the method already described (*Comptes Rendus*, vol. cxxviii., p. 1120; *Bull. Soc. Chim.*, Series 3, vol. xxi., p. 554). The following are the results obtained:—

	M.grm. of iodine per kilo.
Solanea—	
Solanum tuberosum (potato)	0·000
Lycopersicum esculentum (tomato)	0·07
Solanum melongena (egg plant)	0·01
Cucurbitacea—	
Cucumis sativus (cucumber)	0·012
,, ,, (gherkin)	0·00
,, melo (melon)	0·06
Cucurbita maxima (pumpkin)	0·017
Crucifera—	
Raphanus sativus (radish)	0·18
Brassica napus (turnip)	0·24
,, ,, (long radish)	0·16
Raphanus ,, (black radish)	0·00
Chenopodea—	
Beta rapa (beetroot)	0·14
,, cycla (skin of beetroot)	0·38
Spinacia oleracea (spinach)	0·021
Leguminosea—	
Faba vulgaris (bean)	0·14
Phaseolus vulgaris (French beans	0·32
,, ,, (kidney beans)	0·013
Pisum sativum (green peas)	0·084
Liliacea—	
Allium sativum (garlic)	0·94
,, cepa (onions)	0·24
,, porrum (leeks	0·12
Polygonea—	
Rumex acetosa (sorrel)	0·047
Umbellifera—	
Scandix cerfolium (cherville)	0·14
Petroselinum sativum (parsley)	0·00
Daucus carotta (carrot)	0·00
Synantherea—	
Lactuca sativa (lettuce)	0·096
Cichorium intybus (chicory)	0·000
,, augustifolium (endive)	0·000

This table shows that under identical conditions of soil, moisture, and exposure some plants absorb much more iodine than others, and that some, on the other hand, do not absorb a trace.

Unfortunately, my experiments were carried out on too restricted a number of plants for me to be able to definitely observe the numerous conditions which affect this absorption. They have, however, been sufficient to show that certain families, the *Liliacea* and the *Chenopodea*, accumulate much more iodine than certain others, such as the *Solanea* or the *Umbellifera*, for example. We also see that, in the same vegetable family, the absorption of this metalloid varies with each variety; the *Synantherea* and the *Crucifera* give very decided examples.

In a forthcoming paper I propose showing that this phenomenon of specific selection of iodine also takes place in the various organs of the same animal species.—*Bull. Soc. Chim.*, Series 3, xxiii., No. 1.

A NEW METHOD OF METHYLATION.

By MAURICE PRUD'HOMME.

This method depends on the combined action of formic aldehyde and nascent hydrogen in an acid medium. By its means I have been able, by methylation, to transform a certain number of colouring materials and leuco bases, containing groups of NH_2. The equation of the reaction is written, $RNH_2+CH_2O+H_2=RNHCH_3+H_2O$.

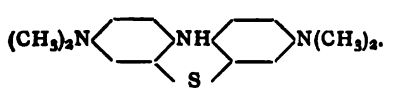
Transformation of Fuchsine into Hexamethylated Violet.—A solution of fuchsine, to which is added hydrochloric acid, zinc powder, and formic aldehyde, is gently heated to 75—80° for about a quarter of an hour. The colourless liquid is filtered, cooled, and oxidised in the presence of acetic acid, with 1 molecule of PbO_2 per 1 molecule of fuchsine. The lead is removed by means of SO_4Na_2, and the colouring matter precipitated by NaCl.

Cotton, mordanted with tannin and tartar emetic, takes a violet colour, only different to hexamethylated violet (hexamethyl parafuchsine) by its slightly bluish tint, which is probably due to the fact that commercial fuchsine is not pure *p*-fuchsine, but a mixture of fuchsines of C_{19} and C_{20}.

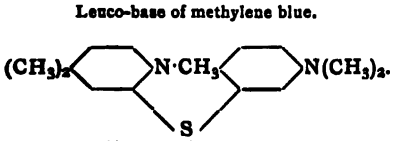
Rhodamine of m-*Aminophenol into Methylated Rhodamine.*—The rhodamine of *m*-aminophenol colours wool, mordanted with tannin, orange. When treated with formic aldehyde, zinc, and hydrochloric acid it becomes methylated. Filtered, the colourless solution gradually regains its colour on exposure to the air. The new rhodamine gives wool, silk, and cotton, mordanted with tannin, the beautiful red shades, of the special tone belonging to the colouring matters of this class.

Thionine into Methylene Blue.—Methylene blue represents tetramethylated thionine (*Lauth violet*). The transformation under the conditions already described is easily effected. Dyed materials, subjected to several successive treatments, re-oxidised in the air, lead to the identification of a new colouring material with methylene blue. When the action of the reagents is pushed further we obtain a second colouring material which dyes cotton treated with tannin and tartar emetic a much greener blue than methylene blue.

It is probable that the methylation, after having acted on the aminogene groups, also takes place with the inside group, NH, of the leuco-base of methylene blue.



Leuco-base of methylene blue.



Methylated leuco-base.

According to Bernthsen, this latter becomes oxidised, giving a blue closely resembling methylene blue, but furnishing a dye of different shades.

Methylene blue, treated directly with CH_2O, Zn, and HCl, gives the same greenish blue colouring materials.

Transformation of Safranines.—We heat a solution of phenosafranine at 1 grm. per litre, in the presence of Zn and HCl, to about 75—80°, until the reduction and decolouration is complete. At this moment the addition of CH_2O instantaneously re-colours the solution a violet-red. A part of this solution, filtered and precipitated by NaCl, will dye cotton mordanted with tannin. The tint is similar to that of Witt's neutral red. If the reaction is prolonged for several minutes the violet-red changes to violet. Finally, after the reaction has lasted about half an hour the colourless, filtered liquid gradually becomes re-oxidised, and takes a blue colour. The colouring material, precipitated by means of NaCl, gives cotton, treated with tannin and emetic, wool, and silk a violet-blue colour.

This blue colour may be an *induline*. It dissolves with an olive colour in SO_4H_2, which becomes a bluish grey on dilution. Generally the safranines give, with SO_4H_2, a green solution, rendered first blue, and then violet by dilution.

Possibly, besides the methylation of the aminogene groups, there may be one produced in the nitrogen of the azonium, by substitution of the hydrogen of the fixed leuco derivative by nitrogen. We have seen above that CH_2O acts as an oxidising agent on the N or the H of the leuco derivative of safranine.—*Moniteur Scientifique*, Feb., 1900, Series 4, xiv.

ON THE ELECTROLYTIC PREPARATION OF PERCHLORIC ACID AND OF ITS SALTS.

By F. FORSTER.

The author has already shown (*Zeit. für Elek.*, ii., p. 270) that when a basic solution of an alkaline iodate is electrolysed a reduction takes place at the anode. This does not occur in the case of the chlorates; that is, if we use a cathode of platinum, lead, copper, zinc, or nickel. By using an iron or cobalt cathode, however, the chlorates can be electrolytically reduced to chlorides. We propose here to deal only with electrolysis in neutral or acid solution; in this case the chlorate is peroxidised into perchlorate—a fact already observed by Stadion and Kolbe.

To examine the influence of various external conditions on the oxidation, we electrolysed 100 c.c. of a solution of known strength between two platinum electrodes of 12 square c.m. surface; the whole being placed in a glass cylinder, hermetically sealed, through the cork of which passed, besides the wires, a glass tube for carrying off any gas disengaged. The gas given off was analysed, as was also that given off concurrently from a gas voltameter placed in the circuit. According to the proportion of oxygen present in the two gases, the percentage of current used for oxidation was calculated. The returns, expressed in percentages of the theoretical amount obtained under various conditions, are given in the accompanying table. The current densities are expressed in ampères per square decimetre.

The solutions of chlorate of potassium and sodium used were neutral; the first contained a little sea-salt, which was transformed into chlorate then perchlorate, without interfering with the phenomenon. The degrees of concentration were chosen in such a way that the solutions of chlorate of soda should be either equivalent to, or four times greater than, that of chlorate of potassium at 5 per cent. The solution of chloric acid was nearly twice that of the chlorate. After electrolysing for a short while, the slightly soluble perchlorate is precipitated from the solu-

		17—20°.						75°.		
Current Density		16·6.		8·3.		4·1.		0·83.	16·6.		8·3.
Duration of experiment		5′.	½ hr.	10′.	1½ hrs.	20′.	3 hrs.	2 hrs. 4 hrs.	5′.	½ hr.	10′.
Returns with a solution of	5 per cent $KClO_3$	75	64	64	65	—	—	33 43	—	—	0·0
	4·2 per cent $NaClO_3$	75	69	63·5	63·5	63	56	— —	—	—	—
	17 per cent $NaClO_3$	89	90	83	88	67	83	50 46	13	8	—
	6·5 per cent $HClO_3$	—	—	79	87	—	—	— —	—	—	—

tion of chlorate of potash at the anode. This salt can also be prepared by adding chloride of potassium to a solution of perchlorate of soda or perchloric acid. The salt thus obtained with perchloric acid loses 45·9 per cent of its weight at a red heat; the salt $KClO_4$ should lose 46·2 per cent.

The table shows that, for currents of too low densities, the return increases with the concentration. From the fact of the feeble solubility of chlorate of potash at a low temperature, and also that in this case we cannot use warm solutions, it is preferable to use chlorate of soda for preparing the perchlorates.

The following are the results obtained with a 50 per cent solution of $NaClO_3$ at the ordinary temperature with a current density of 8·3 ampères per square decimetre; the tension being 4·5 volts at the electrodes when these are 1·5 c.m. apart.

Time from the commencement.	Return, per cent of theory.
10 minutes	86*
5 hours	95
20 ,,	98
24½ ,,	95
27½ ,,	24
29½ ,,	28

* In another experiment we obtained 95 per cent after ten minutes.

To transform 50 grms. of chlorate of soda into perchlorate requires twenty-five hours theoretically with the current used above, viz., 1 ampère. It can be seen that the useful effect of the current and the proportion of chlorate transformed are very considerable. By doubling the current no other result was noticed beyond diminishing the duration of the experiment by half and an increase of electromotive force to 5·2 volts. Towards the end of the experiment the smell of ozone became more and more pronounced.

The solutions of perchlorate obtained by the preceding experiments were treated with the theoretical amount of KCl. We thus obtained in $KClO_3$ 78—85 per cent of the quantity of $NaClO_3$ in solution. The salt obtained retained a small proportion of chlorate. By simply measuring the oxygen given off, we obtained 45·7 and 45·5 per cent, instead of 46·2 per cent which should be given off by the pure salt.

With solutions of $KClO_3$ the results, as we have seen, are not so good. An experiment was made with three litres of a 5·6 per cent solution of the salt, using a platinum anode between two nickel cathodes. In this manner we obtained 82 per cent of the theoretical return, estimated by means of a copper voltameter placed in the circuit; but, when once about half the chlorate was oxidised, the return decreased with great rapidity. The production of perchlorate thus takes place as well in an acid as in a neutral solution. In alkaline solution we have only observed an energetic oxidation at the commencement, which is the stronger as the strength of the current is greater, but which decreases after a certain time. The following series of observations, made on a 50 per cent solution of $NaClO_3$, containing 1 per cent of NaHO, with a current strength of 1 ampère (equal to 8·3 ampères per square decimetre) shows this fact plainly:—

The quantities of oxygen used at the anode were, in percentages of the oxygen produced by the current—

After 1 minute .. .. .. 20 per cent
,, 3 hours .. .. .. 8 ,,

The strength of the current was then increased to 2·3 ampères (equal to 23·3 ampères per square decimetre), and the results obtained were:—

After 10 minutes .. .. 54 per cent
,, 30 ,, ... 53 ,,
,, 1½ hours .. .. .. 44 ,,
,, 4 ,, ... 2 ,,

It does not therefore seem possible, according to our experiments, to obtain perchlorates directly from the electrolysis of chlorides, as the intermediate product—the chlorate—can only be formed in alkaline solution; the alkali being produced by the current.—*Zeitschrift für Elektrochemie*, iv., p. 373.

A REVISION OF THE ATOMIC WEIGHT OF COBALT.*

Third Paper.—The Analysis of Cobaltous Chloride and Oxide.

By THEODORE WILLIAM RICHARDS
and
GREGORY PAUL BAXTER.

The two preceding papers (*Proc. Amer. Acad. of Arts and Sciences*, 1897, xxxiii., 113, and 1898, xxxiv., 351; Chemical News, lxxvii., p. 20, and lxxix., p. 199), describing the earlier part of this investigation, were concerned with the complete analysis of cobaltous bromide. Both the cobalt and the bromine in this compound, as well as the trace of inevitable impurity, were determined with care, and the consistent verdict of all the results pointed toward a value between 58·99 and 59·00 for the number sought. The danger of the existence of an unknown error in a result thus obtained from a single compound is too well known to need emphasis; and with this danger in mind we sought to obtain support from wholly different data. The present paper describes two series of determinations, which, while less satisfactory than the work with the bromide, are nevertheless of corroborative value, and moreover serve to point out some of the pitfalls which ensnared the earlier experimenters.

I. *The Reduction of Cobaltous Chloride.*

Cobaltous chloride was chosen first as the basis of the confirmatory work, both on account of the ease with which it can be prepared and because it can be heated to a much higher temperature than the bromide without danger of vaporisation. These circumstances allow greater surety both in drying and in reducing.

Owing to the comparatively slight volatility of cobaltous chloride, and to the difficulty of obtaining chlorine in a sufficiently pure state, we made no attempt to purify the salt by sublimation. The preparation of cobaltous chloride in aqueous solution was inadmissible on account of the certain presence of oxide in the dried and ignited substance under these conditions; hence the method finally adopted was the ignition of carefully purified purpureo-cobaltic chloride, which may be dried before it decomposes.

A considerable quantity of this compound was prepared by passing air through a strongly ammoniacal solution of

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxv., No. 3, 1899.

cobaltous chloride, and subsequently acidifying the mixture with hydrochloric acid. The precipitate was then re-crystallised six times by solution in pure re-distilled ammonia in a platinum dish and precipitation with re-distilled hydrochloric acid in a Jena glass flask. This latter operation could not be performed in platinum, because of the liberation of small quantities of chlorine. The final precipitate was collected on a pure washed filter and dried. In order to convert the amine into cobaltous chloride it was heated in an air-bath to about 200°, until it was entirely decomposed without fusing. Owing to the fact that the dried salt melts at a temperature very slightly above that at which it breaks up, the heat was applied very gradually. Here again the liberation of traces of chlorine prevented the use of platinum, a porcelain crucible being used to contain the salt. The corresponding purpureo-bromide of cobalt melts at a temperature too near its decomposing-point to have allowed the use of a similar method for preparing cobaltous bromide in the earlier part of this research.

The cobaltous chloride thus prepared, still containing a large amount of ammonic chloride, was now heated in the platinum boat in a rapid current of dry nitrogen and hydrochloric acid gas until ammonic chloride ceased to be expelled. The drying apparatus used in the work upon the bromide was re-filled, and easily altered for this purpose by substituting a strong solution of hydrochloric acid for the bromine and red phosphorus. The hydrochloric acid gas was displaced by pure nitrogen, and this in turn by pure air; and finally the dry cobaltous chloride was transferred to the weighing bottle by means of the usual bottling apparatus.

After having been weighed, the chloride was reduced in hydrogen in the manner already described under the analysis of cobaltous bromide.

During the reduction no trace of *cobaltous* chloride sublimed from the boat, but a slight sublimate of *ammonic* chloride appeared in the tube. The amount of this sublimate was determined by washing out the tube with pure water and Nesslerising the solution, and this amount was subtracted from the original weight of cobaltous chloride. Probably the correction was somewhat too great, for traces of ammonic bromide appeared in every reduction of cobaltous bromide, when no ammonia could have been present in the salt; but the error arising from this source must have been small.

A small quantity of alkaline chloride was found in the residual cobalt, and was determined in the usual way by lixiviating the metal and evaporating the solution. This correction was of course subtracted from the original weight of the cobaltous chloride, as well as from that of the metal.

In the following table of results the specific gravity of cobaltous chloride is assumed to be 2·94, the value given by Joule and Playfair (Landolt and Börnstein, *Tabellen*, 1894, p. 134). This necessitates a correction to the vacuum standard of +0·000262 grm. per grm. of chloride. Cobalt itself has a specific gravity too near that of brass to need any correction of this kind. Cl=35·455.

Observed weight of cobaltous chloride in vacuum	4·16702	2·30588
Observed weight of cobalt in vacuum	1·89304	1·04771
Weight of residue	0·00061	0·00048
Weight of ammonic chloride	0·00158	0·00028
Corrected weight of cobaltous chloride	4·16483	2·30512
Corrected weight of cobalt	1·89243	1·04723
Atomic weight of cobalt	59·053	59·035
Average	59·044	

As the alkali had not been completely removed by the process of crystallisation to which the cobaltamine had been subjected, it is highly probable that some silica also remained which was not removed from the metal by leaching. This probability, together with the fact that the ammonic chloride correction is undoubtedly somewhat too large, seems sufficient to account for the fact that these results are slightly higher than those obtained in the bromide series (58·995). At any rate the evidence of this method is that the atomic weight of cobalt cannot be greater than 59·05, for none of the probable errors would tend to make this result too low.

(To be continued).

A TABLE OF ATOMIC WEIGHTS
OF SEVENTY-FOUR ELEMENTS.*

By THEODORE WILLIAM RICHARDS.

Name.	Symbol.	Atomic weight.
Aluminium	Al	27·1
Antimony	Sb	120·0
Argon	A	39·9?
Arsenic	As	75·0
Barium	Ba	137·43
Beryllium	Be=Gl	9·1
Bismuth	Bi	208·0
Boron	B	10·95
Bromine	Br	79·955
Cadmium	Cd	112·3
Cæsium	Cs	132·9
Calcium	Ca	40·1
Carbon	C	12·001
Cerium	Ce	140·0
Chlorine	Cl	35·455
Chromium	Cr	52·14
Cobalt	Co	59·00
Columbium	Cb=Nb	94·0
Copper	Cu	63·60
"Didymium"	Nd+Pr	142 ±
Erbium	Er	166·0
Fluorine	F	19·05
Gadolinium?	Gd	156·0?
Gallium	Ga	70·0
Germanium	Ge	72·5
Glucinum	Gl=Be	9·1
Gold	Au	197·3
Helium	He	4·0?
Hydrogen	H	1·0075
Indium	In	114·0
Iodine	I	126·85
Iridium	Ir	193·0
Iron	Fe	56·0
Lanthanum	La	138·5
Lead	Pb	206·92
Lithium	Li	7·03
Magnesium	Mg	24·36
Manganese	Mn	55·02
Mercury	Hg	200·0
Molybdenum	Mo	96·0
Neodymium	Nd	143·6
Nickel	Ni	58·70
Niobium	Nb=Cb	94·0
Nitrogen	N	14·045
Osmium	Os	190·8
Oxygen (standard)	O	16·000
Palladium	Pd	106·5
Phosphorus	P	31·0
Platinum	Pt	195·2
Potassium	K	39·140
Praseodymium	Pr	140·5
Rhodium	Rh	103·0
Rubidium	Rb	85·44
Ruthenium	Ru	101·7
Samarium?	Sm	150·0
Scandium	Sc	44·0
Selenium	Se	79·2

* Compiled in April, 1899, from the most recent data.

Name.	Symbol.	Atomic weight.
Silicon ..	Si	28·4
Silver	Ag	107·930
Sodium ..	Na	23·050
Strontium ..	Sr	87 68
Sulphur ..	S	32·065
Tantalum ..	Ta	183·0
Tellurium ..	Te	127·5 ?
Terbium? ..	Tb	160·0
Thallium ..	Tl	204·15
Thorium ..	Th	233·0
Thulium? ..	Tu	170·0 ?
Tin	Sn	119·0
Titanium ..	Ti	48·17
Tungsten ..	W	184·4
Uranium ..	U	240·0
Vanadium ..	V	51·4
Ytterbium ..	Yb	173·0
Yttrium ..	Yt	89·0
Zinc	Zn	65·40
Zirconium ..	Zr	90·5

NOTE.

Since the appearance of this table in 1898, the Committe of the German Chemical Society (Messrs. Landolt, Ostwald, and Seubert) have made their interesting report upon the subject, and have invited the chemists of the world to join them in deciding upon one standard to be used everywhere. The fulfilment of this very desirable end must necessarily be a matter of many months; hence the present table is re-published this year in accordance with the original plan. It is to be distinctly understood that the re-publication is not in any way an attempt to compete with or to forestall the International Committee; it is merely an expression of opinion, which may be of temporary service. The fact that none of the other recent tables follow the accepted scientific usage concerning significant figures seems to afford an additional reason for reprinting this one.

The investigations of the past year have pointed to a change in four values given in the table of 1898. Calcium is made 40·1, instead of 40; for recent experiments (as yet unpublished) in this laboratory indicate that last year's estimate was too low. Neo- and praseodymium were oddly transposed by their discoverer, and the more accurate values of Jones (*Am. Chem. Journ.*, 1898, xx., 345; CHEMICAL NEWS, lxxvii., 280) and others are substituted. Lastly, Denher's (*Journ. Am. Chem. Soc.*, 1898, xx., 555; compare Clarke, *Ibid.*, 1899, xxi., 200; CHEMICAL NEWS, lxxix., 195) careful investigation upon selenium seems to show that this element has a higher atomic weight than was formerly supposed to belong to it. For the present a compromise number, 79·2, is recorded above.—*Proceedings of the American Academy of Arts and Sciences.*

Royal Institution.—The Right Hon. Rayleigh, F.R.S., will not deliver the second of his course of Lectures on "Polarised Light" at the Royal Institution on Saturday afternoon next (the 10th inst.), owing to the sudden death of his mother, the Dowager Lady Rayleigh. There will be no Lecture on Saturday afternoon.

International Exhibition at Canea, 1900.—On the 11th April next there will be opened by Prince George of Greece, "The First International Exhibition at Canea," the capital of Crete. No effort will be spared by the Government to make it a success; the Austrian, Italian, Greek, and Russian steam navigation companies will endeavour to make the passage to Crete as convenient and frequent as possible, and it is considered that a profitable market can be opened up in the island for a variety of goods. All particulars as to cost of space, &c., can be obtained from Mr. Arthur Gobiet, Prague, Carolinenthal, Bohemia, Austria. The Exhibition will remain open until the 7th of May, 1900.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 15th, 1900.

Sir HENRY ROSCOE, LL.D., F.R.S., Vice-President, in the Chair.

(Concluded from p. 103).

*21. "*The Combination of Sulphur Dioxide with Oxygen.*" By EDWARD JOHN RUSSELL and NORMAN SMITH.

The authors have found that when a mixture of sulphur dioxide and oxygen acts on certain oxides, in addition to the absorption of the sulphur dioxide, part of the sulphur dioxide and oxygen combine, forming sulphur trioxide, this being apparently due to the "surface action" of the oxide. The extent of this "surface action" varies with the nature and physical conditions of each oxide. No sulphur trioxide was ever found unless a simultaneous absorption of sulphur dioxide occurred; when manganese peroxide and sulphur dioxide, dried by phosphorus pentoxide, were brought together, no absorption took place, nor was any sulphur trioxide produced on the addition of dried oxygen.

If a mixture of dried sulphur dioxide and oxygen be passed over well-dried platinised pumice heated to 400—450°, very little sulphur trioxide is formed, and the drier the materials the less is the combination.

*22. "*Notes on the Estimation of Gaseous Compounds of Sulphur.*" By EDWARD JOHN RUSSELL.

In this paper volumetric methods of analysis are given which have been found to work satisfactorily in the estimation of sulphur dioxide, hydrogen sulphide, carbonyl sulphide, and carbon disulphide in gaseous mixtures. It is found that, except in the case of sulphur dioxide, absorption methods are not reliable; as a rule, by far the most satisfactory method is to explode with oxygen. In this explosion, sulphur dioxide and sulphur trioxide are both produced, the amount of the former being determined directly, and that of the latter calculated from the contraction observed after explosion. Sulphur dioxide is best estimated by absorption with a pellet of lead peroxide, since this substance, if freshly washed and not too dry, exerts but little of the surface action which tends to cause some of the sulphur dioxide to combine with part of the oxygen to form sulphur trioxide.

*23. "*The Influence of the 'Nascent State' on the Combination of Dry Carbon Monoxide and Oxygen.*" By EDWARD JOHN RUSSELL.

The author has studied several reactions in which carbon monoxide and excess of oxygen were brought together in the flame of an explosion, one or both being in the nascent condition. Combustion was rarely complete, and the amounts of carbon monoxide left unburnt were found to be very similar to those left unburnt in Dixon's experiments (*Trans.*, 1896, 784), in which dried mixtures of carbon monoxide and oxygen were fired by adding carbon disulphide and oxygen, and sparking, although in the present experiments one of the substances (carbon monoxide) was in the nascent condition, whilst in Dixon's experiments it was not. It seems, therefore, that the nascent condition has no great effect in promoting combination between carbon monoxide and oxygen, although the high temperature produced by a violent reaction has a very considerable effect.

The substances used as sources of nascent carbon monoxide were carbonyl sulphide and nickel carbonyl; and for nascent oxygen, chlorine monoxide and chlorine peroxide.

It was discovered incidentally that mixtures of pure carbonyl sulphide and excess of oxygen are not exploded by an electric spark. If, however, a slight amount only

of impurity is present, an explosion takes place, but combustion is not complete, carbonyl sulphide remaining unburnt in large quantity, along with carbon monoxide, produced by the decomposition of some of the carbonyl sulphide. If the impurity is greater, the combustion is complete.

*24. "*Asymmetric Optically Active Tin Compounds. Dextromethyl ethyl-n-propyl Tin Iodide.*" (Preliminary Note). By WILLIAM JACKSON POPE and STANLEY JOHN PEACHEY.

Hitherto no substance containing an atom of tin attached to four different groups has been obtained, owing to the lack of general methods for their preparation.

The authors find that compounds of the type—

$$SnX^{I}X^{II}X^{III}X^{IV}$$

can be readily prepared by aid of the following series of reactions from the trimethyl tin iodide described by Ladenburg and Cahours:—

(1). $2Sn(CH_3)_3I + Zn(C_2H_5)_2 = 2Sn(CH_3)_3(C_2H_5) + ZnI_2$.
(2). $Sn(CH_3)_3(C_2H_5) + I_2 = Sn(CH_3)_2(C_2H_5)I + CH_3I$.
(3). $2Sn(CH_3)_2(C_2H_5)I + Zn(C_3H_7)_2 = 2Sn(CH_3)_2(C_2H_5)(C_3H_7) + ZnI_2$.
(4). $Sn(CH_3)_2(C_2H_5)(C_3H_7) + I_2 = Sn(CH_3)(C_2H_5)(C_3H_7)I + CH_3I$.

Externally compensated *methylethyl-n-propyl tin iodide*, $\begin{matrix}CH_3\\C_2H_5\end{matrix}\!\!>\!Sn\!<\!^{C_3H_7}_{I}$, the final product of reaction (4) is a faintly yellow oil boiling at 270° without decomoposition; it is almost insoluble in water, and its vapour exercises unpleasant effects on the mucous membrane.

On agitating the iodide with a warm aqueous solution of a molecular proportion of silver dextrocamphorsulphonate, and evaporating the filtered liqnid, a copious deposit of *dextromethylethyl-n-propyl tin dextrocamphorsulphonate*, $Sn(CH_3)(C_2H_5)(C_3H_7)(C_{10}H_{15}OSO_3)$, was obtained; the salt is only sparingly soluble in cold water, and crystallises in lustrous square plates melting at 125—126°. In dilute aqueous solution it has the molecular rotation $[M]_D = +95°$, so that for the monobasic radicle, $\begin{matrix}CH_3\\C_2H_5\\C_3H_7\end{matrix}\!\!>\!Sn-$, $[M]_D$ is about +45°.

On evaporating the mother-liquor from which this salt had separated, further deposits of the same salt were obtained until all the water had been expelled, and the authors have been unable to isolate a salt of the lævo-base; this behaviour seems due to the conversion of the lævo-base into the dextro-base by continued racemisation in the following manner:—The solution of externally compensated base with dextrocamphorsulphonic acid deposits a portion of its dextro-base as the sparingly soluble salt; the excess of lævo- over dextro-base remaining in the solution racemises as evaporation proceeds, a further portion of dextro-base separates as salt, and racemisation of the residue again proceeds. This case is the first of the kind recorded, and affords powerful support to Le Bel's hypothesis respecting the mobility of alkyl groups of low molecular weight in quaternary ammonium salts.

On treating the freshly prepared cold aqueous solution of the dextrocamphor sulphonate with potassium iodide solution, *dextromethylethyl-n-propyl tin iodide* separates as a faintly yellow oil; the highest specific rotation of the iodide which has yet been observed is $[\alpha]_D = +23°$ in ethereal solution. The rotation was, however, very variable, and, under conditions not yet understood, the separated iodide was optically inactive. This behaviour is due to racemisation.

It should be noted that the discovery noted above, that racemisation, due to intramolecular mobility, may cause rapid and complete conversion of an externally compensated base into one optically active isomeride, reopens the question of the space confiuration of quadrivalent sulphur compounds which, in the light of previous knowledge, seemed to have been determined by our former results (*Proc.*, 1900, xvi., 12).

Having now shown that quadrivalent asymmetric tin compounds can exist in enantiomorphously related configurations possessing optical activity, it follows that the space configuration of quadrivalent tin compounds is of the tetrahedral type already established for carbon compounds, and substances of the type of tin tetrachloride must now be regarded as true atomic compounds. Having proved that asymmetric pentavalent nitrogen compounds (*Trans.*, 1899, lxxv., 1127) also give rise to optical activity in the same way that van 't Hoff and Le Bel proved it for carbon, it may be concluded that all enantiomorphous compounds must be optically active.

In Group IV. of the periodic table the non-metal, carbon, is at the top, whilst the metal, tin, is near the bottom; since both can give rise to optical activity in their asymmetric compounds, it is highly probable that all the elements in Group IV. lying between these are capable of acting as centres of optical activity. Should this hold for the whole of Group IV., and as it has been shown to hold for nitrogen, the first member of Group V., all the elements of the latter group may be expected to act as optically active centres in appropriate compounds. Compounds of the types—

$$RX^{I}X^{II}X^{III}X^{IV} \text{ and } QX^{I}X^{II}X^{III}X^{IV}X^{V}$$

should therefore be optically active, R being an atom of any element in Group IV., and Q an atom of any element in Group V.

The application of the reactions now described to other elements is in progress, and the authors hope shortly to communicate results obtained with quadrivalent lead compounds.

25. "*Note on the Refraction and Magnetic Rotation of Hexamethylene.*" By SYDNEY YOUNG, D.Sc., F.R.S., and EMILY C. FORTEY, B.Sc.

The specimen of hexamethylene obtained by one of the authors from Galician petroleum was found to contain a small quantity of a heptane. The hexamethylene was freed from the paraffin by fractional crystallisation, and its refraction and magnetic rotation were determined by Dr. W. H. Perkin, sen. The results obtained are given in the paper.

26. "*Apiin and Apigenin.* Part II. *Note on Vitexin.*" By A. G. PERKIN.

By the action of nitric acid on apigenin under varied conditions, four nitro-derivatives have been obtained: *mononitro-apigenin*, $C_{15}H_9O_5(NO)_2$, orange-yellow needles, m. p. about 302°, somewhat soluble in alcohol; *trinitro-apigenin* (*a*), $C_{15}H_7O_5(NO_2)_3$, yellow needles, m.p. about 296°; *trinitro-apigenin* (*b*), yellow needles, m. p. 245—246°; and *tetranitro-apigenin*, $C_{15}H_6O_5(NO_2)_4$, almost colourless needles, m. p. 243—244°. With the exception of the first mentioned, all dissolve with difficulty in the usual solvents. In preparing the mononitro-compound, *dinitro-p-hydroxybenzoic acid* was obtained as a decomposition product, a further proof that apigenin does not contain a catechol group (*Trans.*, 1897, lxxi., 805). Tetranitro-apigenin appears to be identical with the compound $C_{15}H_6O_5(NO_2)_4$, obtained from vitexin (*Trans.*, 1898, lxxiii., 1026), and, as the decomposition products of both colouring matters are identical, vitexin must be a derivative of apigenin. The formulæ $C_{21}H_{20}O_{10}$ and $C_{21}H_{13}O_{10}(C_2H_3O)_7$ are now assigned to vitexin and acetylvitexin, and it is suggested that vitexin is *a stable glucoside of apigenin*, and that a like constitution may also apply to scoparin (*Proc.*, 1899, xv., 123), which, according to this view, is a glucoside of a luteolin monomethyl ether.

Apiin, $C_{27}H_{32}O_{16}$, the glucoside of apigenin occurring in *Apium petroselinum*, is not converted by dilute nitric nitric acid into nitro-apigenin, but into *nitroapigetrin*,

$C_{21}H_{21}O_{11}NO_2$, a yellow crystalline powder, very sparingly soluble in the usual solvents. This is the nitro-compound of a new glucoside, apigetrin, which differs from apigenin in containing but one sugar group. By prolonged digestion with dilute hydrochloric acid, it yields the above mononitro apigenin.

27. "*The Yellow Colouring Principles of various Tannin Matters.*" VII. By A. G. Perkin.

The yellow colouring-matter of the leaves of *Arctostaphylos uva-ursi* (bearberry) and *Hæmatoxylon Campeachianum* (logwood) is *quercetin*, and this is accompanied by a second substance, probably *myricetin*, to which the green colour of its alkaline solutions are due. *Gallotannic acid* occurs in some quantity in the latter leaves. The leaves of *Rhus metopium* contain *gallotannin*, *myricetin*, and a trace of *quercetin*, but the stem of this plant, unlike *R. cotinus* and *R. rhodanthema*, is devoid of colouring-matter. The sparing solubilities of acetylmyricetin and dibromoquercetin have been employed for the separation of myricetin and quercetin. The leaves of *Robinia pseudacacia* contain a feeble colouring-matter, *acacetin*, $C_{16}H_{12}O_5$, which yields an *acetyl* derivative, $C_{16}H_{10}O_5(C_2H_3O)_2$, colourless needles, m.p. 195—198°, and on fusion with alkali *phloroglucinol*, *p-hydroxybenzoic acid*, and a trace of *protocatechuic acid*. Acacetin contains one methoxyl group, on removal of which a colouring-matter, $C_{15}H_{10}O_5$, results, having the reactions of apigenin. It is thus probably an *apigenin monomethyl ether*. The leaves of *Myrica gale* and *Coriaria myrtifolia* contain respectively *myricetin* and *quercetin*. Although a relationship frequently exists between the tanning and the colouring-matters of the same plant (*Trans.*, 1897, lxxi., 1138) there is no rule on this point, for exceptions are somewhat numerous.

28. "*Note on the Bromo-derivatives of Camphopyric Acid.*" By J. Addyman Gardner.

An atom of hydrogen in camphopyric acid may be readily replaced by bromine, by the action of the halogen in the presence of phosphorus. Two acids of the formula $C_9H_{13}BrO_4$ are thus obtained, and are provisionally named α- and β bromocamphopyric acid. These acids may be prepared either by treating camphopyric acid with phosphorus pentachloride, and subsequently warming with bromine, or by mixing the acid with amorphous phosphorus and adding the proper amount of bromine. In either case the product is poured into cold water, when part of the acid separates in the solid state and part is extracted with ether from the solution. The α- and β-acids may be separated by repeated crystallisation from benzene.

If only a small amount of water is used to decompose the acid bromides, bromocamphopyric anhydride is also obtained.

α-Bromocamphopyric acid separates from benzene or chloroform as a very light, white, crystalline powder, melting at 167° with previous softening. It is very moderately soluble in hot benzene and chloroform, but only slightly in cold; it is also very soluble in water, ether, acetone, and ethyl acetate. It is a dibasic acid, and forms very soluble sodium, barium, and ammonium salts.

α-Bromocamphopyric anhydride, $C_9H_{11}BrO_3$, may be obtained by treating the acid in the usual way with acetyl chloride or acetic anhydride. It crystallises readily from benzene in well-defined tabular crystals which melt at 226—227°. These crystals are very soluble in ether, acetone, and ethyl acetate.

β-Bromocamphopyric acid is more soluble in benzene than its isomeride, and is obtained, though in very small quantity, from the mother-liquors of the α-acid. It is a white crystalline substance melting at 207—208°, quite different in appearance from its isomeride. Its barium salt is soluble, and has the formula $Ba(C_9H_{12}BrO_4)_2,4H_2O$; this acid has not, however, been obtained in sufficient quantity for a more complete investigation.

The same products are obtained when camphoic acid is similarly treated with phosphorus pentachloride and bromine.

α-Hydroxycamphopyric acid, $C_9H_{13}(OH)O_4$. If α-bromocamphopyric acid is heated for some time with a 10 per cent solution of potash, then acidified with hydrochloric acid and extracted with ether, an oily acid is obtained which for the most part solidifies on standing. If this be boiled with benzene the oily portion dissolves, and a white solid is left, which, on crystallisation from water, may be obtained in well-defined, colourless, transparent crystals which melt at 206—207°. This substance is a dibasic acid, and has the formula $C_9H_{14}O_5$. Its sodium and barium salts, which are very soluble in water, were analysed. It is only very slightly soluble in benzene, chloroform, and carbon bisulphide, but dissolves easily in ethyl acetate and in hot water; the oil obtained along with it is apparently an unsaturated acid, but has not yet been obtained in a state of sufficient purity for investigation. These substances are being prepared in quantity, and the author hopes before long to be able to bring before the Society a more complete account of their properties and reactions.

PHYSICAL SOCIETY.

Special Meeting, March 2nd, 1900.

Prof. G. Carey Foster, F.R.S., Vice-President, in the Chair.

This Meeting was held, by invitation of Professor Callendar, in the Physical Laboratory of University College.

Dr. F. G. Donnan read a paper on "*The Relative Rates of Effusion of Argon, Helium, and other Gases.*"

The introduction to this paper contains a short account of the work which has been done on the effusion of gases. This is followed by a theoretical investigation of the subject, upon the assumption that the ideal gas laws are obeyed and that the back pressure never rises above a certain fraction of the internal pressure. This gives rise to formulæ which are different from the square root of the density law of Graham. The formula derived from Hugoniot and Reynolds' work gives the ratio of the times of effusion of two gases whose specific heat ratios are 1·408 and 1·67, equal to 1·06 times the square root of the ratio of the densities. The constant derived from Parenty's work is 1·084. The theory therefore indicates that argon should effuse faster than would be calculated from Graham's law. The gases used were oxygen, hydrogen, nitrogen, carbon-monoxide, carbon-dioxide, cyanogen, argon, and helium, and they effused through small holes pierced in platinum foil. When the holes are large compared with molecular dimensions, the phenomenon is one of efflux on a small scale. In the actual experiments this was the case, although the holes were sufficiently small to cause appreciable viscosity effects. By employing two or more observations in conjunction with the relative viscosities of the gases used, an apparatus constant was determined which allowed these effects to be eliminated. The observations showed that argon effused 3½ per cent faster than as calculated from the densities alone. This agrees qualitatively with theory, and affords a confirmation of the high specific heat ratio of argon. Hydrogen, oxygen, and carbon-monoxide effuse in the manner predicted by the theory for gases having the same or nearly the same specific heat ratios. Carbon dioxide, when compared with oxygen, appears to effuse 1 per cent faster than as calculated from the densities. This result is not in accordance with the adiabatic theory of the efflux of ideal gases. The results for helium are not uniform, but show that its behaviour is unlike that of argon, a result not foreseen by the theory. If account be taken of the deviation of ordinary gases from the ideal laws, it is

possible to obtain an expression for the efflux which contains a correction term involving the constant K of the Joule-Thomson effect. The sign of this correction term shows that a real gas will effuse more rapidly or more slowly than an ideal gas of equal density and specific heat ratio, according as K is positive or negative. The suggestion is made that possibly the anomalous results obtained with carbon-dioxide and helium may be thus explained. The deviations of the observed results from the results calculated for an ideal gas are, in the case of carbon-dioxide, in qualitative agreement with the theory proposed. In the case of helium they would be so if that gas possesses a negative K.

Lord Rayleigh congratulated the author and pointed out that in the case of very small apertures the gas laws might not be obeyed. The ratio of the dimensions of the aperture to the length of the mean free path determined this, and not the ratio of aperture to molecular dimensions.

Prof. Ramsay and Prof. Everett expressed their interest in the work.

Dr. Donnan thanked Lord Rayleigh for his correction, and stated that the apertures used were about 1/50 m.m. in diameter.

Mr. E. C. C. Baly read a paper on "*The Distillation of Liquid Air and the Composition of the Gaseous and Liquid Phases.*"

From the experiments described in this paper the author has drawn curves showing the relation between the composition of the gas evolved by boiling liquid air and the temperature, and between the composition of the liquid and the temperature, both at constant (atmospheric) pressure. These curves enable the temperature of boiling liquid air to be at once accurately determined by means of an analysis either of the liquid or of the gas evolved. The measurements of temperature were made with a Callendar compensated constant pressure hydrogen thermometer. The correction for the contraction of the glass bulb was determined by measuring directly the linear expansion of glass between $-190°$ and $20°$ C. This was found to be 0·0000073, Regnault's measurement between $0°$ and $10°$ being 0·0000085. The values for the boiling-points of oxygen and nitrogen agree fairly well with those given by Olszewski and Estreicher. Boiling nitrogen has a great tendency to superheat. This can be obviated by passing a rapid current through the boiling liquid, or by dropping in pieces of copper. There does not appear to be any connection between the ratio of the vapour-pressures and the composition of the gaseous phase in the distillation of oxygen and nitrogen at constant pressure. It is proposed to investigate the distillation at constant temperature.

Prof. Ramsay drew attention to the uses of liquid air for carrying on researches at low temperatures. It is non-explosive, easy to work with, and is easily kept either by means of a vacuum jacket or by surrounding it with cotton-wool.

Prof. Callendar referred to the question of superheating, and stated that the constant-pressure thermometer was more accurate than the constant volume one for measuring low temperatures.

A paper on "*The Reversibility of Galvanic Cells*," by Mr. T. S. Moore, was read by Dr. Lehfeldt.

In these experiments the reversibility of cells such as the Daniell and the Clark, which are assumed to be reversible, was tested by allowing the cell to send a current and by sending a current through the cell. The E.M.F.'s of the cells were determined by means of a Crompton potentiometer, and from the E.M.F.'s on open and closed circuits the internal resistance of the cells were calculated.

Prof. S. P. Thompson asked if experiments had been made upon Leclanché cells, where the products of the action escape.

Dr. Lehfeldt said that experiments were not made upon these cells, because they were known not to be reversible.

A paper on "*The Damping of Galvanometer Needles*," by Mr. M. Solomon, was postponed until the next meeting.

The meeting then adjourned until March 9th.

NOTICES OF BOOKS.

Handbook of Practical Hygiene. By D. H. Bergey, A.M., M.D. Easton, Pa.: The Chemical Publishing Company. 1899. Pp. 164.

The practical hygiene of to-day has grown up from the observations and experience gained in many lands by various people. Most of these observations were made in the present century, as practically nothing had till then been done in advance of the rough methods of the ancients.

Most of the important work on water supplies in connection with certain diseases has been done in the last few decades, though much earlier the value of a sufficient air supply had been recognised by Howard, in regard to the high death rate in prisons. Besides plenty of air and a good water supply, modern sanitary science also takes note of the character of the soil with regard to dampness and porosity, or draining power.

Until the appearance of this volume there has been no short and concise laboratory guide to the subject published in English, though there are several treatises that cover the whole field, and a few handbooks in German.

The author may therefore reasonably expect a welcome to his work, which also includes, besides what we have already referred to, the elementary sanitary analysis of foods, and a chapter on ventilating and heating.

Lessons in Physical Chemistry ("Leçons de Chimie Physique") delivered at the University of Berlin, by J. H. van 't Hoff. Translated from the German by M. Corvisy. Part II. Static Chemistry. Paris: Librairie Scientifique, A. Hermann. 1899. Pp. 162.

The Lectures which form the subject of this volume were delivered by the author in the collegiate year 1897–98. Though forming the second part of the work, of which Dynamic Chemistry is Part I., this volume may also be considered an independent book, treating of the methods to which we owe what knowledge we at present possess of the size, structure, and grouping of molecules—methods which have given rise to the modern conception of the constitution of matter.

This volume is divided into three portions: Part I. on molecular weight and polymerism; Part II. on molecular structure (isometry, tautometry); Part III. on molecular grouping (polymorphy).

In volume I., besides the conception of the molecule, no hypothesis was needed on the nature of matter; in this volume the atomic hypothesis, on which depend the complex problems of structure, is introduced; while in the third volume, which is to follow, will be brought forward the still very obscure question of the relations between properties and composition, the whole forming a book of the highest value and interest.

A New Synthesis of Nitric Acid.—S. Tanatar.—The author has prepared nitric acid by reacting with sulphate of hydrazine dissolved in water on a 3·3 per cent solution of chloride of nitrogen in benzene. The two solutions are continuously shaken up together for two hours. The aqueous layer is then neutralised with soda, decanted, and supersaturated; a quarter of the liquid is distilled. The return is from 5 to 6·5 per cent. The best results are obtained by adding a little 10 per cent soda from time to time, until the alkaline reaction remains constant. The return is then 36 per cent, and could no doubt be still further increased.—*Berichte*, xxxii., p. 1399.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 7, February 12, 1900.

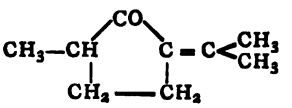
Researches on the Uric Series.—M. Berthelot.—The study of the uric series has of late years undergone a complete change. E. Fischer's theories on the fundamental radicle—purine—have caused the necessity of a further thermo-chemical examination of this series and extension of the interesting results obtained by M. Matignon. For an exhaustive study, this research would necessitate the preparation of thirty or forty new compounds discovered by M. Fischer. Without extending the work to this extent, the author found it possible to establish a certain number of essential relations, by choosing a series of typical substances for experiment. These were the glycollic and lactic nitriles, the indol derivatives, and xanthine. These compounds were used to establish two important thermo-chemical problems; especially the thermic effects of the addition of oxygen and those of the methylic substitution in the uric series.

Dispersion of the Radium Rays in a Magnetic Field.—Henri Becquerel.—In a recent paper the author shows that the deviable portion of the radium rays are dispersed in a magnetic field. The deviated pencil being composed of radiation of which the trajactories are rays of different curvature, and of which the absorption across different screens varies with the nature and position of these screens. The author has improved the original experimental conditions, and obtained some results which are of assistance in the further study of these phenomena. Measurements have been taken with screens of paper, aluminium, mica, glass, platinum, copper, and lead.

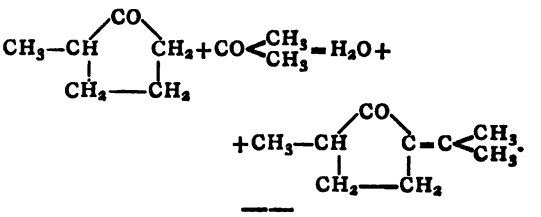
Synthesis of Campholic Acid by means of Camphoric Acid.—A. Haller and G. Blanc.—A. Baeyer and V. Williger recently produced campholide by oxidising camphor by means of a solution of potassium persulphate in sulphuric acid. The authors have further effected the change of campholide into campholic acid. The campholide is first transformed into bromocampholic acid by saturating a solution of lactone in acetic acid with hydrobromic acid. The bromine derivative is precipitated little by little, the precipitation being completed in about twenty-four hours. This is collected on a filter, washed with acetic acid, and dried in a vacuum; this body is in the form of white crystals melting at 177°, and is identical with the substance which is obtained directly from camphor.

Composition of the Essence of Sandal Wood from the West Indies.—M. Guerbet.—The author was able to isolate the following definite compounds from sandal wood:—(1) Two sesqui-terpenic carbides, $C_{15}H_{24}$. Sandalenes α and β, which are colourless oily liquids, with slight odour. Sandalene α boils at 252—252·5°, having a density of 0·9134 at 0°; it is lævo-rotatory $\alpha_D = -13·98°$. Sandalene β boils at 261—262°, has density 0·9139 at 0°, and rotatory power $\alpha_D = -28·55$. (2) A mixture of sesqui-terpenic alcohols, $C_{15}H_{26}O$, of rotatory power varying from $\alpha_D = -9·4°$ and $\alpha_D = -25·3°$, and corresponding absolutely to the preceding carbides. (3) An aldehyde of formula $C_{15}H_{24}O$, sandalal, a colourless liquid boiling at 180° at 40 m.m. pressure. (4) An acid of formula $C_{15}H_{24}O_2$, sandalic acid, obtained by oxidising sandalal with chromic acid in acetic solution. (5) Teresandalic acid, $C_{10}H_{14}O_2$, which crystallises from alcohol in large colourless prisms melting at 157°. (6) The most volatile portions of the essence, of strong odour, the author has not obtained in a state of purity.

Synthesis of the Phorone of Camphoric Acid.—L. Bouveault.—As a result of the work of W. Kœnigs and A. Eppens, the following formula was arrived at to express the phorone of camphoric acid:—



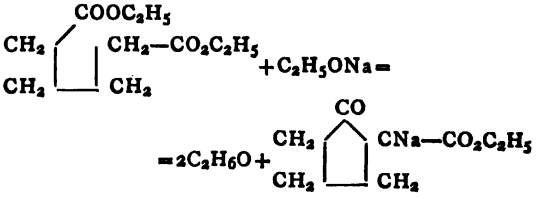
this being the generally accepted formula. The author synthesises this compound by condensing α-methylcyclopentanone—a substance he has previously synthesised—with ordinary acetone; the action appears to take place according to the equation—



Bulletin de la Société Chimique de Paris. Series 3, Vol. xxi., No. 23.

On the Constitution Formula of Camphor.—L. Bouveault.—Not suitable for abstraction.

Synthesis of Cyclopentane Derivatives by means of Adipate of Ethyl.—L. Bouveault.—Most chemists now agree in making camphoric acid a cyclopentane derivative. The author thought it would be of interest to know what were the simplest bodies attached to this series; for this purpose he made use of a method described by M. Dieckmann, who condensed adipate of ethyl by means of ethylate of sodium according to the equation—



He did not isolate this β-ketonic ether, but he showed its existence by forming a copper salt. The cyclopentanone carbonate of ethyl is the simplest of the cyclic β-ketonic ethers, and was prepared by the author almost quantitatively as follows:—To one molecule of adipate of ethyl dissolved in its own weight of toluene is added one molecule of clean sodium, and the whole kept at 120—140° for four hours. The product, after cooling, is added to an excess of a 10 per cent solution of acetic acid. The oily layer contains toluene, cyclopentanone carbonate of ethyl, and a little unchanged adipate. These bodies can be separated by precipitating the ketonic ether in the form of a copper salt by means of a solution of acetate of copper. This result can be obtained more simply by shaking up the oil, first with an excess of 15 per cent potash, and then with strong potash; the mixed solutions are acidulated with acetic acid, the oil separated, washed with ether and carbonate of sodium, then, after driving off the ether, distilling in vacuo, when the desired ether is obtained in a state of perfect purity. Cyclopentanone-carbonate of ethyl is an oily, colourless liquid boiling at 113° under 22 m.m. pressure, $d = 1·0976$ at 0°. It has an acetylacetic odour, and answers to the formula $C_8H_{12}O_3$.

On the Natural Cyclisation of Citronnellal.—H. Labbé.—The author left a quantity of citronnellal in a nearly full flask for about two months. At the end of one month the odour was considerably modified. After sixty days the characteristic odour of citronnellal had com-

pletely disappeared, and was replaced by a strong smell of menthol. After shaking up with bisulphide to remove the unaltered citronnellal it was rectified at the ordinary pressure. Nearly the whole passed over at 205—210°. Cyclic transformation was immediately suspected, but it could not have been effected by means of an acid agent; further, the alcohol obtained was free and not etherified. To identify this alcohol it was oxidised with a chromic mixture, and an oil boiling at 210° was obtained. The ketone thus obtained was transformed into a semicarbazone in hydroalcoholic solution of chlorhydrate of semi-carbazide and acetate of sodium. After three days it was precipitated by water, and finally purified by crystallisation in ligroïn. Its fusing point is 171—172°.

On the Formation of Citryl and Citronellylsulphites of Barium.—H. Labbé.—5 grms. of the normal sodic bisulphitic derivative of citral was dissolved in 150 c.c. of distilled water, at the same time 3 grms. of $BaCl_2$ were dissolved in 15 c.c. of water; on adding these two solutions together a voluminous precipitate is formed. This is filtered after half an hour, and the precipitate dried on a porous plate; it weighed 1·3 grm. Two hours afterwards, another precipitate was deposited, weighing in the same manner 0·35 grm.; finally, after forty-eight hours, a third precipitate was formed which also weighed 0·35 grm., the weight of the three thus being 2·0 grms. Theory requires 5·89 grms. In a second experiment 1 grm. of citronellylsulphite of sodium in 30 c.c. of water and 0·8 grm. of $BaCl_2$ in 8 c.c. of water were taken; the precipitate weighed 0·93 grm.; theory required 1·18 grm. This shows that when the bisulphitic derivative is sufficiently pure the precipitate is much more abundant, and exceeds 80 per cent of theory, whereas when not pure it may be as low as 17 per cent.

MISCELLANEOUS

The Tertiary Aromatic Amines.—C. Haeusermann and E. Bauer.—For examining the action of nitrous acid on the tertiary aromatic diamines, the authors have prepared some tetraphenylphenylene-diamines by the reaction of the dichlorbenzenes and diphenylamine soda. With *p*-dichlorbenzene they obtained two isomerous tetraphenylphenylene-diamines, one of them, *α*, purified by crystallisation in acetone, takes the form of brilliant colourless leaves, fusible at 199—200°, the other, *β*, is more soluble in acetone, and melts at 127—129°. The former, treated in acetic solution by nitrate of soda, gives a nitrated derivative, crystallised in brick-red needles fusible at 167—168°; the second, under the same conditions, gives a product crystallising in glacial acetic acid in brilliant yellowish-brown prisms, fusible at 185—186°. The formation of two isomers in the above case is no doubt due to the fact already observed by Istrati that *p*-dichlorbenzene becomes changed at a high temperature, into *m*-dichlorbenzene. While working with *o*-dichlorbenzene, the authors prepared tetraphenyl-*o*-phenylenediamine, a crystalline powder, fusible at 133—134·5°.—*Berichte*, xxxii., p. 1912.

Preparation of *α*-Dinitrodimethylaniline.—J. Pinnow.—*α*-Dinitrodimethylaniline is obtained by means of dinitrochlorbenzene and dimethylaniline, or, according to Mertens, by means of dimethylaniline and dilute nitric acid: the best method is that of Schuster, which consists of operating with dilute nitric acid in the presence of a large quantity of sulphuric acid. The return did not, however, reach more than 90 to 110 per cent of the weight of dimethylaniline used, while theoretically we ought to obtain 174·4 per cent. The mother-liquors from the preparation of the *α*-dinitrodimethylaniline contain a fairly large quantity of *o*-dinitrodimethylaniline. The author has determined the character of this base, which is in the form of a reddish oil, by preparing its picrate, which crystallises in fine prisms, fusible at 102—103·5°: *o*-amidodimethylaniline, obtained by the reduction of the *o*-nitro by means of Zn+HCl, is an oil distilling at 217·5° under a pressure of 715 m.m., having the smell of menthol. Fe_2Cl_3 gives a cherry-red colour to its alcoholic solution. Its hydrochlorate occurs in white prisms, fusible at 184—186°; its picrate in leaves fusible at 138—140°. The acetyl derivative appears in fine white needles, fusible at 72—73°. Heated for half-an-hour with double its weight of acetic anhydride, to 160°, it gives N-*α*-dimethylbenzimidazol.—*Berichte*, xxxii., p. 1666.

Deviation of the Becquerel Rays in the Magnetic Field.—F. Giesel (*Wied. Ann.*, No. 12, 1899) shows that these rays are deviated by magnets. A preparation of polonium was the source of the rays. Radium also showed the same phenomenon but not to so high a degree. —*Am. Journ. Sci.*

Preparation of Pure Tertiary Anilines and Tetra-alcoylised Aromatic Diamines.—J. Pinnow.—The author found in the use of warm ammonia a simple means for the preparation of the tertiary anilines, the tetra-alcoylised aromatic diamines, the dimethylamido phenols, the dimethylamidobenzoic acids, &c., from their halogeno-alcoylised products of addition; it is only necessary to obtain an alcoylation as advanced as possible, and to remove the impure tertiary bases from the product of the reaction before using the ammonia. Some observations were made during this research which recall the work of Bischoff and Menschutkin; we know that these chemists found that certain groups interfered with the complete alcoylisation of the base, probably on account of their stereo-chemical character. Thus the addition of a halogen-alcoyl to the tertiary anilines in which the ortho-position relative to an amido-group is occupied, offers certain difficulties; it is therefore necessary to modify the conditions of the reaction according to the case presented. The number of basic or acid groups also plays an important role in the separation of the halogeno-alcoyl by means of ammonia; a temperature of 110—140° suffices in the case of monamines and of amido-carbonic acids, for example, while for diamines and the amidophenols it is necessary to heat to above 140°, preferably to 180—190°. The time of heating is generally an hour at 180—190°, and two or three hours at 130—140°; the ammonia is transformed into mono-, di-, or trimethylamine. Experiments were made with the three phenylene-diamines, with benzidene, ortho-amido-phenol, *p*-amidobenzoic acid, and with the naphthylamines.—*Berichte*, xxxii., p. 1401.

The Preparation of Crystallised Calcium.—Henri Moissan.—Crystallised calcium can be prepared by two methods. To obtain it in a state of purity the author made use of the property, hitherto unknown, of dissolving it in liquid sodium kept at a dull red heat. On cooling, the calcium crystallises out, and, by treating the metallic mass with absolute alcohol, brilliant white hexagonal crystals of pure calcium remain. 600 grms. of anhydrous crystallised iodide of calcium are placed, with 240 grms. of sodium, in an iron crucible of 1 litre capacity; this is closed with a screw-down cover, and heated to dull redness for an hour. On cooling, the crucible is found to contain a layer of melted salt of a blue colour, covered by a cake of metallic sodium. This sodium, broken up to fragments of about 1 c.c., is added to 500 c.c. of anhydrous absolute alcohol, kept at 0° in a 1 litre flask. The return of calcium is about 50 per cent of the theoretical amount. Similar crystals of calcium can be obtained by the electrolysis of melted iodide of calcium at a dull red heat. The negative electrode is of pure nickel and the positive of graphite. Under these conditions a white melted, or crystalline, metal is obtained. —*Bull. Soc. Chim.*, Series 3, xxi., Nos. 20—21.

On the Percarbonates.—S. Tanatar.—The author has obtained percarbonates by reacting with H_2O_2 on Na_2CO_3 or K_2CO_3. These percarbonates, heated to 100°, do not give off oxygen, while those prepared by the electrolytic method by Hansen and Constam give off both CO_2 and O. Percarbonate of soda should correspond to the formula, $Na_2CO_4 + 1\frac{1}{2}$ aq. This salt is gradually decomposed in aqueous solution, into H_2O_2 and Na_2CO_3; when an acid is added there is a disengagement of CO_2, and the solution retains H_2O_2, which can, however, be extracted with ether. The author, to make certain that he had not to do with a mixture of Na_2CO_3 and H_2O_2, determined the heat of decomposition of the salt by nitric acid, and he found the number of calories (7·210—7·260) higher than that required by the decomposition of Na_2CO_3 (5·33 cals.). Percarbonic acid is thus a weaker acid than carbonic. He has not succeeded in obtaining pure percarbonate of potassium. With metallic salts the sodium salt gives precipitates, which easily decompose with the evolution of oxygen, so that it has not been possible to prepare the corresponding percarbonates.—*Berichte*, xxxii., p. 1544.

MEETINGS FOR THE WEEK.

MONDAY, 12th.—Society of Arts, 8. (Cantor Lectures). "Photography of Colour," by E. Sanger Shepherd.

TUESDAY, 13th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

—— Society of Arts, 8. "English Furniture," by Lasenby Liberty.

WEDNESDAY, 14th.—Society of Arts, 8. "Continuation School Work in Rural Districts, by H. Macan, M.A., F.C.S.

THURSDAY, 15th.—Royal Institution, 3. "Recent Excavations at the Argive Heræum (in Greece)," by Charles Waldstein, Litt D., Ph.D., L.H.D.

—— Chemical, 8. "The Vapour Densities of Dried Mercury and Mercurous Chloride," by H. Brereton Baker, M.A. "The Preparation of Pure Hydrobromic Acid" and "A New Sulphide of Arsenic," by A. Scott, D.Sc., F.R.S. "The Action of Iodine on Alkalis," by R. L. Taylor. "The Interaction between Sulphites and Nitrites," by Edward Divers, D.Sc., F.R.S., and Tamemasa Haga, D.Sc. "New Polysaccharides —Manno-galactan and Lævulo-mannan," by Julian L. Baker and Thomas H. Pope.

FRIDAY, 16th.—Royal Institution, 9. "Pictorial Historical Records," by Sir Benjamin Stone, M.P.

SATURDAY, 17th.—Royal Institution, 3. "Polarised Light," by The Rt. Hon. Lord Rayleigh, M.A., D.C.L., &c.

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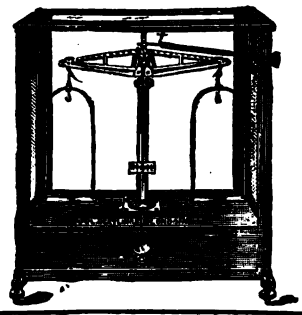

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THE CHEMICAL NEWS.

VOL. LXXXI., No. 2103.

RADIATION FROM RADIO-ACTIVE BODIES.

By HENRI BECQUEREL.

THE author obtained, from M. and Mme. Curie, a specimen of polonium nitrate which was nearly as radio-active as radium, whether measurements were made on the increase of conductivity of air or on the action on a photographic plate.

The radiation from this substance appeared to be unaffected by a magnetic field, so differing from radium radiation.

The polonium is placed between the poles of an electro-magnet, in a magnetic field of intensity 4000, and above it a photographic plate, not enclosed in a black envelope on account of the absorption of polonium rays by black paper. After some minutes a symmetric impression will be found on developing the plate, and this impression is the same whether the electro-magnet is excited or not.

If radium is substituted for polonium, the experimental conditions remaining the same, a non-symmetric impression is found on the plate when the magnet is excited, showing that the rays have been shifted in the direction of the current, producing the field.

In a previous paper the varying penetration of polonium and radium rays with regard to different substances has been discussed. From consideration of the experiments described in this paper and previous work, the author arrives at the conclusion that the radium rays resemble cathode rays to a certain extent, but have not all their properties. For example, if, in a magnetic field which has been rendered practically uniform, a small quantity of radium is placed on a photographic plate enveloped in black paper, horizontal and parallel to the field, then at right angles to this is placed a second plate, whose position with regard to the radiant source and the pole pieces varies in different experiments. After some moments, on developing the horizontal plate, curves are found, which have been described in a previous paper, whilst on the vertical plate is an intense impression limited by a spiral in the direction of the current which produced the field.

These experiments allow of the measurements of the deviation in a magnetic field.—*Comptes Rendus*, 1899. vol. cxxix., No. 26.

THE ACTION OF MERCURY ON IODIDE OF METHYLENE.

By V. THOMAS.

WHILE the organo-metallic derivatives in which the metallic bonds are saturated by monovalent radicals have long been studied, such is not the case with the organo-metallic derivatives, which can be foretold theoretically by substituting for several hydrogen atoms of a carbide, di-, tri-, and tetra-valent metallic atoms.

In the hope of isolating some metallic compounds of olefiant gas, Messrs. Wanklyn and Von Than studied the action of mercury and zinc on iodide of ethylene, but they were unable to arrive at the wished-for goal. In 1880 Sakurai took up their experiments, but substituted chloride, bromide, and even chloroiodide of ethylene, for the iodide; unfortunately he was no more successful than his predecessors. He had, however, the idea of replacing the ethylenic halogen compounds by iodide of methylene. On leaving iodide of methylene and metallic mercury in contact in a sealed tube, in the presence of a small quantity of mercurous iodide, he observed that after four or five days the contents of the tube were transformed into a yellowish-white mass, which further took a red colour after a longer time. On opening the tube no trace of gas was noticed. The product of the reaction consisted principally of two compounds, one of which dissolved easily in iodide of methylene; the other, on the contrary, was quite insoluble in this body. The soluble compound consisted, according to this authority, of the derivative $CH_2I.HgI$. The insoluble derivative was not analysed.

More recently M. Moissan (*Bull. Soc. Chim.*, Series 3, vol. xx., p. 1010) has prepared the carbide of aluminium, C_3Al_4. On decomposing this body by water, he effected a remarkable synthesis of methane:—

$$C_3Al_4+6H_2O=3CH_4+2Al_2O_3.$$

From this it follows that this carbide of aluminium corresponds to the formula $Al\equiv C-Al=C=Al-C\equiv Al$, and constitutes the first organo-metallic derivative known, in which each bond of the metal is not saturated by a monovalent carbonated radical.

The research I have undertaken had for its object to determine the behaviour of the different metals with iodide of methylene; I thought it would be first of advantage to repeat Sakurai's experiment.

Action of Mercury on Iodide of Methylene in the Cold.—By leaving $Hg.Hg_2I_2$ and CH_2I_2 in contact for several days I obtained the compounds described by Sakurai, but the mercurous iodide, contrary to what was said by that chemist, does not appear to play any *rôle* at all. In fact, the reaction is quite identical if we proceed without this salt.

In a future paper I shall return to the compounds thus obtained.

Action of Mercury on Iodide of Methylene when Warmed.—If, instead of proceeding at the ordinary temperature, we use heat, the reaction becomes more rapid; but after a certain temperature gaseous products are given off. I have studied the reaction at about 200°. For this purpose 40 grms. of mercury were heated with 22 grms. of iodide of methylene to between 190° and 200° for ten or twelve hours in a sealed tube. After the reaction the tube contains large crystals of mercuric iodide, some red and others yellow. The liquid has completely disappeared.

The gases given off are collected over mercury, and submitted to the following tests:—

1. Treatment by potash; no absorption.
2. Treatment by pyrogallic acid; no absorption.
3. Treatment by acid cuprous chloride; no absorption.
4. Treatment by bromine; no absorption.
5. Treatment by fuming sulphuric acid; no absorption.
6. Treatment by ammoniacal cuprous chloride; no absorption.

Eudiometric analysis gave the following figures:—

Taken as sample	2·27 c.c.
Oxygen added	8·22 ,,
Volume after sparking	6·1 ,,
,, ,, absorption by KOH ..	3·1 ,,
Remainder not absorbed by pyrogallic acid	0·44 ,,
Oxygen not used	2·75 ,,

As we find, from preliminary tests, that the gases formed belong to the series of saturated carbides, the analytical results lead us to consider them as a mixture of methane and ethane in the proportion of $CH_4+2C_2H_6$. In fact, the reaction of the combustion is as follows:—

$$CH_4+2C_2H_6+18O=5CO_2+8H_2O.$$

This reaction shows that 1 volume of carbide ought to give 2·6 volumes of steam and 1·6 volumes of carbonic

acid. The figures found by analysis are $H_2O = 2{\cdot}4$ vols., and $CO_2 = 1{\cdot}6$ vols. — *Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 2.

A NEW METHOD FOR THE ESTIMATION OF CYANIDE OF MERCURY.

By E. VINCENT.

The estimation of cyanide of mercury offers certain difficulties, owing to the great stability of the salt. It may be effected in the following manner:—

A. Precipitation of the mercury in the state of sulphide in the cyanide solution, to which an ammoniacal solution of nitrate of zinc has been added, and estimation of the mercury after solution in *aqua regia*.

B. Estimation of the cyanogen in the form of cyanide of silver, in the solution from which the mercury has been removed.

This method gives very good results, but it requires long and careful manipulation. It appeared to me to be desirable to find a more rapid method, without sacrificing efficiency.

The principle of the method is as follows:—When cyanide of mercury is heated with soda-lime, the mercury is set free in the metallic state, and the nitrogen is converted into ammonia. By collecting and weighing the mercury, and, on the other hand, collecting the ammonia in titrated sulphuric acid, we can simultaneously estimate the mercury and the nitrogen.

As can be seen, the method consists entirely in combining the two processes, the one for the estimation of the mercury (action of lime on mercurial compounds), and the other for the estimation of the nitrogen in certain organic compounds (Will and Varrentrapp's method).

Experiment has shown that it can be perfectly well applied to the case in question.

I have experimented on samples of perfectly pure cyanide, and I have also made comparative tests with the ordinary method and the soda-lime method.

In the following table I give some of the results obtained:—

		Found.	Calculated for $Hg(CN)_2$.
Sample No. 1.	Ordinary method ..	Hg = 78·5 %	Hg = 79·3 %
		N = 10·99 „	N = 11·11 „
	Soda-lime method ..	Hg = 78·3 „	—
		N = 10·91 „	—
Sample No. 2.	Ordinary method ..	Hg = 78·3 „	—
		N = 10·91 „	—
	Soda-lime method ..	Hg = 78·4 „	—
		N = 10·89 „	—

The method is very rapid, and the above results show that it is perfectly applicable.

In practice cyanide of mercury is easily obtained in a pure state, and does not often need to be tested. Such, however, is not the case with commercial oxycyanides, and it is especially in this direction that the above described method will be useful.

I have estimated a certain number of samples of oxycyanide, and I obtained extremely variable results. One sample gave results very close to those of pure oxycyanide; some have a composition which decidedly resembles that of cyanide of mercury, while others appear to be mixtures of various proportions of cyanide of mercury and mercuric oxide.

These variable results obtained with the oxycyanides show that the substitution of the oxycyanide for the cyanide, in the sterilising of surgical instruments, is quite possible.

It might perhaps be preferable to use simply cyanide of mercury, with carbonate or borate of soda added, as proposed by M. Denigès.—*Journ. de Pharm. et de Chim.*, Series 6, vol. x., No. 12.

ACTION OF HYDROCHLORIC ACID GAS UPON SULPHATES, SELENATES, TELLURATES, AND PHOSPHATES.*

By RAYMOND W. TUNNELL and EDGAR F. SMITH.

Experiments have been made at various times in this laboratory upon the action of the gaseous haloid acids on metallic oxides and sulphides. Frequently new methods of separation have resulted. These have proved very satisfactory in every respect, from the standpoint of the analyst.

In the following paragraphs we give additional data collected from the same field, and trust that they may not be devoid of interest to the reader.

Knowing that selenic and telluric acids could not be expelled from their salts by hydrochloric acid gas we endeavoured to ascertain whether, if a sulphate were mixed with one or both of the preceding salts, it would be possible to drive out the selenic or telluric acid and leave the sulphate intact. Accordingly a perfectly dry mixture of a sulphate and a selenate was exposed to the action of well-dried hydrochloric acid gas. The selenic acid was not only expelled, but also varying amounts of sulphuric acid were driven out. Thus, two trials were made with definite amounts of the salts. The results are these:—

Potassium sulphate taken.	Potassium selenate taken.	Sulphur remaining in the salt acted upon.
Grm.	Grm.	Per cent.
0·4323	0·4567	33·59
0·3338	0·1583	33·51

In brief, about 65 per cent of the sulphur originally contained in the potassium sulphate was expelled in the presence of the selenate. When a tellurate was mixed with the sulphate and the two subjected to the action of hydrochloric acid gas, not only was the tellurium driven out from its combination, but the sulphur also in amounts which varied with the working conditions. The original idea therefore of effecting a separation was abandoned.

We next exposed pure, dry potassium sulphate to the influence of the haloid acid. A weighed amount of the salt was introduced into a porcelain boat, placed in a combustion tube of hard glass, through which the gas was conducted in the cold. It was very soon observed that the salt had increased very considerably in weight (as much as 12 per cent). After a time the boat was removed and the residue in it analysed. Thus, 1·0896 grms. of potassium sulphate after exposure for three and one-half hours to the gas, in the cold, weighed 1·1162 grms.—an increase of 0·0266 grm. The salt was dissolved in water, and the hydrochloric acid in it determined. It equalled 0·0268 grm., or a difference of 0·0002 grm. from the observed increase in weight. This product (an addition product evidently) maintained its composition when exposed to 130° in a drying oven. It did not lose in weight on standing over soda-lime, in a vacuum desiccator, for fifteen hours.

When dry potassium sulphate was heated in hydrochloric acid gas from 200° to a dull red heat, an examination of the water in the receiving vessel showed the presence of sulphuric acid, while the substance remaining in the boat after this treatment weighed more

* Contribution from the John Harrison Laboratory of Chemistry. From *The Journal of the American Chemical Society*, vol. xxi., No. 10.

than the original substance. It gave an acid reaction to litmus, and decolourised a red phenolphthalein solution. As much as 19 per cent of the sulphuric acid in the salt was expelled in this way. The quantity of acid driven out was greater in the presence of a selenate or tellurate, equalling in several instances 75 per cent.

This deportment of sulphuric acid confirms an early observation of Heusgen (*Ber. d. Chem. Ges.*, 1876, 1671), who found that "Kalium sulfat wird weder in der Kälte noch bei gewöhnlicher Temperatur von Salzsäure angegriffen . . . . bei 360 liess sich in dem vorgelegten Wasser schon eine wägbare Menge Schwefelsäure nachweisen, bei dunkler Rothgluth erfolgt jedoch die Zersetzung *fast* quantitativ in Chlormetall und freie Schwefelsäure." The gas evidently attacks the salt in the cold. This is proved by the formation of the addition product. We never observed a complete elimination of the sulphuric acid. It was not, indeed *fast* "*quantitative*." And, Prescott (CHEM. NEWS, xxxvi., 178), trying the action of aqueous hydrochloric acid upon metallic salts, observed that upon evaporating a grm. of sodium sulphate with 4·035 grms. of aqueous hydrochloric acid, containing 1·251 grms. of hydrochloric acid, he obtained only 0·070 grm. of sodium chloride, and 0·807 grm of undecomposed sulphate remainsd.

The question as to how the sulphuric acid is expelled from its salt is worth consideration. The thought first suggested was that the addition compound, K_2SO_4+HCl, is produced when the gas acts upon the salt, and this upon the application of heat decomposes, forming potassium chloride and acid potassium sulphate, the latter being changed by the same heat into—

$$2KHSO_4 = K_2SO_4 + H_2O + SO_3.$$

If this were true then the addition compound, which in theory was formed at first, when heated alone in dry air should give up its sulphuric acid in part at least, and it should be found in the water of the receiver. Several trials were made. Sulphuric acid was not found in the receiver, but instead about 86 per cent of the hydrochloric acid originally present with the potassium sulphate was discovered to be there. After numerous experiments of various sorts we concluded that this remarkable expulsion of sulphuric acid from one of its stable salts is to be attributed to mass action. This is emphasised by its more complete expulsion when a selenate or a tellurate is present.

It is a well-established fact that phosphoric acid is not expelled by the gaseous haloid acids from its salts.

A mixture of dry sodium tellurate and dry sodium pyrophosphate was exposed to the action of hydrochloric acid gas. Care was taken that the temperature did not become so intense as to melt the mass in the boat. When this occurs the telluric acid is apparently reduced very rapidly. In the analysis, which will be introduced, the tellurium was merely expelled. No attempt was made to estimate its amount. The residual phosphate, after solution in water, was invariably examined for tellurium, but it was not detected. The phosphoric acid was determined in the usual manner.

Analyses.

	Phosphorus present in the $Na_2P_2O_7$.	Amount of sodium tellurate taken.	Phorphorus found as $Mg_2P_2O_7$.
	Grm.	Grm.	Grm.
1.	0·1030	0·1247	0·1026
2.	0·1197	0·1632	0·1199
3.	0·1096	0·0832	0·1094

Sodium selenate was next mixed with sodium pyrophosphate. Much less time was required for the volatilisation of the selenic acid than for that of the telluric acid. The temperature was gradually raised to 450° C. It was not allowed, nor was it necessary, to go beyond this point.

Analyses.

	Phosphorus present in the $Na_2P_2O_7$ used.	Sodium selenate.	Phosphorus found as $Mg_2P_2O_7$.
	Grm.	Grm.	Grm.
1.	0·0728	0·1763	0·0729
2.	0·1057	0·2017	0·1054
3.	0·1198	0·1416	0·1199

The preceding results with phosphates and tellurates and those with selenates and phosphates leave no doubt as to the complete separation of their respective acids, in the manner indicated. The method is free from all disturbing factors and may be executed without difficulty.

NOTES ON THE ANALYSIS OF DYNAMITE AND GELATIN-DYNAMITE.

By F. W. SMITH.

I.

THE analysis of dynamite is not often described in text-books, and the following notes, gleaned from some years' experience, may be of interest to those who have occasion to investigate such products.

A simple case is that of a dynamite containing nitro-glycerin, sodium nitrate, wood-pulp, and basic magnesium carbonate or kieselguhr. The following process of analysis is recommended as giving satisfactory results:—Weigh out 10 grms. on a pair of watch-glasses, place in a desiccator over sulphuric acid, and leave for at least five days. The loss in weight is called moisture. Weigh about the same amount into a Gooch crucible and extract with pure ether. A drop of the ether is allowed to evaporate on a piece of tissue paper from time to time, and the extraction continued until the paper no longer tastes of nitro-glycerin. Dry the residue in an air-bath at about 80° C. for two to three hours, and weigh. The loss minus the moisture is called nitro-glycerin. In the case of powders containing only nitro-glycerin and kieselguhr, this extraction is considerably more difficult, and it is advisable to remove the dynamite from the Gooch crucible to a small beaker and stir up with ether, washing the residue back when free from nitro-glycerin. After extracting the nitro-glycerin, the nitre is extracted with cold water, and after the water ceases to extract anything it is displaced by acetone with the object of drying the residue without affecting the starch if any be present. The loss in weight is called sodium nitrate, potassium nitrate being very rarely used. The residue from the water extraction is ignited until free from carbon. The loss is wood-pulp, flour, bran, &c. I have found no satisfactory method for separating these, and rely on testing with iodine for the presence of starch as an indication of the presence of flour, &c.

If no earthy absorbents have been used, the ash after ignition is not likely to be over one-tenth per cent of the whole sample. If the ash is considerable it is extracted with hydrochloric acid, and the residue dried. The loss, expressed in percentages, is subtracted from the percentage of loss on ignition. The loss on ignition minus the loss on extraction with hydrochloric acid is called wood-pulp, &c., while twice the latter is entered as basic magnesium carbonate. This method saves a quantitative determination of the magnesia in the extract, and rests on the fact that the basic magnesium carbonate employed as an absorbent loses practically one-half its weight on ignition. The extract should, however, be examined for lime, &c.; a small amount of iron may come from the kieselguhr. If any residue be left after extracting with hydrochloric acid it is examined under the microscope for kieselguhr, mica, &c., or it may be a small amount of venetian red used for colouring the powder.

The following analysis will serve to illustrate the methods;—

No. 515.	Per cent.	Per cent.
Moisture	1·3	—
Nitro-glycerin	39·6	39·6
Sodium nitrate	46·8	47·1
Wood-pulp	9·5	—
Basic magnesium carbonate ..	1·8	—
Kieselguhr	1·0	—
	100·0	

The loss on ignition was 10·40 per cent, and on extraction with hydrochloric acid 0·90 per cent. A quantitative determination of the magnesia, calculated as $Mg(OH)_2.3MgCO_3.3H_2O$, gave $1\frac{8}{10}$ per cent. The portion used for moisture determination may be used to check the extractions.

Lunge's nitrometer may be used to advantage as a check on the analysis. About ½ grm. of the dynamite is weighed into a small weighing bottle and about 10 c.c. of pure sulphuric acid poured over it. It is then allowed to stand from twelve to eighteen hours. In this time the whole usually goes into solution. In the cold the reaction is perfectly calm and no nitrogen is lost. It is then introduced into the nitrometer and decomposed as usual. The result is most conveniently expressed in cubic centimetres of nitric oxide per grm. On the sample mentioned above three determinations in the nitrometer gave 237·0, 234·5, and 236·5 c.c. of nitric oxide per grm. 39·6 per cent of nitro-glycerin gave 116·9 c.c., 46·8 per cent of sodium nitrate gave 122·9 c.c. of nitric oxide per grm. respectively; total, 239·8 c.c. The sodium nitrate used is generally 96 to 98 per cent pure; assuming 97 per cent reduces the calculated c.c. to 236·1. The nitrometer work shows that in this case the ether-soluble portion contains nothing but nitro-glycerin, the water-soluble portion nothing but sodium nitrate and its usual slight impurities. Eight minutes agitation in the nitrometer is sufficient for almost all cases.

II. *Gelatin-dynamite.*

The gelatin-dynamites are considerably more difficult to analyse. The following analysis may be selected as typical:—

No. 478.	Per cent.
Moisture	0·4
Nitro-glycerin	33·7
Sulphur	1·9
Resin	7·0
Sodium nitrate	54·0
Gun-cotton	1·1
Wood-pulp	1·9
	100·0

The sample for moisture is weighed out as before. A filter cone (C. S. and S.) is dried and weighed in a weighing bottle and about 15 grms. of the powder weighed into it. This is extracted in a Soxhlet apparatus with chloroform. The loss includes nitro-glycerin, sulphur, and resin. Sometimes the powder melts together in the hot chloroform, and in that case it must be taken from the filter and broken up in a beaker with cold chloroform until the bulk of the nitro-glycerin is dissolved. After extracting with chloroform the residue is extracted with cold water to remove the sodium nitrate, dried and weighed, then extracted with acetone, and the loss on extraction with acetone called gun-cotton. The still remaining portion consists of wood-pulp and earthy absorbents which are treated as above described. Sodium nitrate is appreciably soluble in acetone, and should, on that account, be removed before the gun-cotton. It is often recommended to extract with acetone first and precipitate the gun-cotton with chloroform. This method gives results which are too high, because the sodium nitrate dissolved by the acetone is also precipitated along with the gun-cotton. Allowance for this can, however, be made by incinerating the gun-cotton and calculating the ash as sodium nitrate. The second and following extractions can conveniently be made in the filter cone, setting it in a test-tube with perforated bottom. The filter is finally incinerated in a platinum dish with the residue, allowance being made for its ash content, which in one case was 11·4 m.grms.

The chloroform solution is evaporated almost to complete absence of chloroform and taken up with glacial acetic acid. The precipitated sulphur is washed into a weighed Gooch crucible, and washed further with a little strong alcohol to remove the resin. The nitro-glycerin is determined by weighing a suitable portion of the powder (in this case $1\frac{3}{10}$ grms.) into a small beaker. Ether is then poured on, and the powder worked over with a platinum spatula. The ether is several times renewed, being poured on a filter and collected in a glass evaporating dish. The ethereal solution is allowed to evaporate spontaneously in a cool place, the dish being covered with tissue paper. After twelve hours the residue can be washed into the nitrometer with acetic acid, care being taken not to use too much acetic acid, which retards the reaction of decomposition, while if too little be used the reaction may be extremely violent. Even with the greatest care in evaporation some nitro-glycerin is lost, and the results are usually about 1 per cent low.

The calculated c.c. of nitric oxide per grm. on this analysis, assuming the gun-cotton to contain 11·9 per cent of nitrogen, are 99·5 for the nitro-glycerin, 141·7 for the sodium nitrate, and $2\frac{1}{10}$ for the gun-cotton; total, 243·3. Two determinations in the nitrometer gave 243·8 and 242·7. As the sodium nitrate was certainly not pure, this result shows that the figure entered for nitro-glycerin must be too low. Assuming the sodium nitrate to be 98 per cent pure gives 138·9 c.c., and this, plus 2·10 c.c. for the gun-cotton, subtracted from 243·2, total by analysis, gives 102·2 c.c. for the nitro-glycerin, corresponding to 34·6 per cent. Of course, if necessary, the water-soluble portion could be isolated and estimated in the nitrometer. The amount given in the analysis, 33·7 per cent, was determined by analysing the ether-soluble portion in the nitrometer and calculating the nitric oxide obtained as $C_3H_5N_3O_9$. The resin in this case was recognised by its odour, and estimated by difference. An attempt to determine it in alcoholic solution, in presence of nitro-glycerin, by titration with alcoholic potash, failed because nitro-glycerin by itself is decomposed by alcoholic potash, decolorising phenolphthalein solution. Some so-called flameless dynamites contain ammonium nitrate. The ammonia is estimated by distillation from aqueous solution and calculated as nitrate.

The two examples given cover the great bulk of dynamites and gelatin-dynamites used in the United States, but many other mixtures, often of a very fanciful nature, are occasionally encountered, some of them exceedingly difficult if not impossible to analyse. The methods of analysis given in Guttmann's "Manufacture of Explosives," vol. ii., are mainly reliable as far as they go, while some of Sanford's methods are likely to lead to disappointment. His separation of nitro-glycerin from camphor is especially difficult to understand, since nitro-glycerin and carbon disulphide mix completely on the addition of camphor.

It is hoped that this paper will provoke discussion which may lead to the adoption of uniform methods of analysis by the chemists who have to deal with this class of products.

III. *Various Notes.*

The nitrometer is conveniently set by the use of pure potassium nitrate. Exactly ½ grm. is weighed into a weighing bottle, covered with sulphuric acid, and allowed to stand until the solution becomes clear. The nitric oxide from this is brought into the measuring tube, and the amount of air in the reduction tube, which should also contain a few drops of water, is varied until the gas in the measuring tube shows 110·5 c.c. with the air in the reduction tube at 100 c.c. The use of potassium nitrate

eliminates a large number of errors, such as those of reading the barometers, thermometers, &c. The potassium nitrate may be purified by dissolving it in the least possible quantity of cold water and precipitating with an equal volume of pure alcohol. Three times is generally sufficient, and the absence of chlorides and sulphates may be taken as a criterion of purity. Sodium nitrate, made by dissolving sodium in alcohol and neutralising with dilute pure nitric acid, may be used as a check on the potassium nitrate. Leaky stopcocks are a frequent source of annoyance in working with the nitrometer. The stopcock of the measuring tube may be tested each time by allowing it to stay open for an instant after connecting it to the decomposing bulb by means of a thick-walled rubber tube and allowing the mercury reservoir to be at its lowest position. The mercury should not sink in the measuring tube. This assures the tightness of everything from the lower side of the stopcock in the measuring tube to the upper side of the stopcock in the decomposing bulb. The stopcock in the decomposing bulb may be tested by analysing the standard potassium nitrate, first with the level of the mercury in the reservoir about 6 inches above that in the decomposing bulb, and secondly about 6 inches below. If the first analysis shows too low a result, and the second too high, it points conclusively to a leak. A person having much nitrometer work to do must familiarise himself with the grinding of stopcocks, as they rarely leave the manufacturers' hands in a perfect condition. Grease for the stopcocks is made from vaseline with the addition of Japan wax and rosin, the proportions being varied in summer and winter to get a product of suitable hardness and consistency.

The refraction method for the valuation of Chili nitre gives results from 1 to 1½ per cent higher than those obtained by using the nitrometer and calculating the nitric oxide obtained as sodium nitrate. This is due to the almost unvarying presence of potassium nitrate, and the occasional presence of potassium perchlorate. The latter may be determined by fusing at a low temperature in the presence of powdered cupric oxide. The increase of chlorides after fusion is calculated as potassium perchlorate. The following is a complete analysis of one sample:—

No. 471.	Per cent.
Moisture	2·2
Insoluble	0·1
Magnesium sulphate	0·2
Magnesium chloride	0·1
Sodium chloride	0·4
Potassium nitrate	3 6
Sodium nitrate	93 4
Potassium perchlorate	0·0
	100·0

The refraction method gave 96·8 per cent of sodium nitrate by difference.—*Journal of the American Chemical Society*, xxi., No. 5.

THE SPECIFIC GRAVITY OF CÆSIUM.

By A. E. MENKE.

The specific gravity of cæsium was determined many years ago by Setteler and given as 1·88. He, however, worked with relatively small quantities of the metal (0·6 and 1·1 grm.), as his method of preparation did not allow him to obtain cæsium in quantity. I have recently, in collaboration with Prof. Hugo Erdmann of Halle, made cæsium on a large scale, and it seemed to me advisable to re-determine the specific gravity.

The cæsium for this purpose was purified by several re-distillations in hydrogen, and then examined spectroscopically. The metal was weighed in hydrogen and then under liquid paraffin, giving the following results as the specific gravity referred to water:—

No. 1.	2·4001
,, 2.	2·4004
,, 3.	2·3996
,, 4.	2·4002
,, 5.	2·3998
,, 6	2·4001
Mean	2·40003

In each case not less than 4 grms. of metal were employed, and the proper correction made for hydrogen weighing.

These results change the atomic volume of cæsium from the figure usually given (70·6) to 55·3, which does not make the drop in the atomic volume curve from cæsium to barium as abrupt as heretofore. It, however, affects the symmetry of the vertical column. The specific gravity of rubidium is probably not correct. I expect to re-determine it shortly.

The following parts of the atomic volume tables illustrate the points in question:—

Table of Atomic Volumes with Sp. Gr. of Cæsium 1·88.

K	45·4	Ca	25·4	Sc	17
Rb	56·2	Sr	35	Y	24·8
Cs	70·6	Ba	36·5	La	22

Table of Atomic Volumes with Sp. Gr. of Cæsium 2·40003.

K	45·4	Ca	25·4	Sc	17
Rb	56·2	Sr	35	Y	24·8
Cs	55·3	Ba	36·5	La	22

These experiments confirm Beketoff's results on the atomic volume of cæsium.—*Journal of the American Chemical Society*, xxi., No. 5.

A REVISION OF THE ATOMIC WEIGHT OF COBALT.*

Third Paper.—The Analysis of Cobaltous Chloride and Oxide.

By THEODORE WILLIAM RICHARDS and GREGORY PAUL BAXTER.

(Continued from p. 113).

II. *The Reduction of Cobaltous Oxide.*

At this point experimental difficulties in the preparation of the chloride, together with the belief that silica could not be completely removed from the salt without introducing platinum, led to a search for a more satisfactory method. In the hands of the early experimenters upon the atomic weights of nickel and cobalt, reduction of the monoxide yielded good results—results less satisfactory in the case of cobalt than in that of nickel, however, on account of the greater difficulty in preparing the monoxide in a pure state.

Russell (*Journ. Chem. Soc.*, 1863, [2], i., 51), the first investigator to work with cobaltous oxide, found that the higher oxides of cobalt were reduced to the monoxide by heating to a high temperature in air, but that re-oxidation of the monoxide took place while it was cooling. He showed this by igniting in air a crucible containing the black oxide and cooling the crucible suddenly by plunging it into cold water. Under these conditions a film of black oxide was formed on the surface only, while the lower

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxv., No. 3, 1899.

portions remained in the form of brown monoxide. Evidently, to prevent re-oxidation it was only necessary to keep the oxide away from contact with oxygen. The method adopted by Russell was to heat the oxide in a current of inert gas until constant weight was established, and then to reduce the compound in hydrogen, thus obtaining an indirect ratio between oxygen and cobalt.

This method of procedure, which was adopted by all subsequent experimenters on the oxide, seemed open to two serious disadvantages. The first, the difficulty of preparing nitrogen or carbon dioxide free from traces of oxygen, is one which can be surmounted, although not easily. The possibility that small quantities of liberated oxygen may become mechanically entangled in the oxide, in such a way as not to be speedily removed by the current of gas, is a danger even less easily avoided. This oxygen would, of course, re-unite with the monoxide upon cooling, thus making its weight too great.

As an alternative to heating in a current of inert gas, ignition in a vacuum suggested itself as offering distinct advantages, since neither of the dangers just mentioned would be incurred. For this purpose a glazed porcelain tube of 28 m.m. internal diameter, similar to that used for fusing silver in a vacuum, was employed (Richards and Parker, *Proc. Amer. Acad.*, 1896, xxxii., 63; Chemical News, lxxv., 173). The tube was closed as usual with Hempel hollow brass stoppers and heated in a perforated Fletcher furnace by means of a large blast lamp, while an ordinary Sprengel air-pump served to exhaust the gases from the tube.

Very pure cobalt, which had been prepared by various methods during the previous work, was converted into the oxide as follows:—From the solution of the metal in pure nitric acid the pink granular cobaltous hydrate was precipitated by adding an excess of pure freshly re-distilled ammonia, and subsequently digesting the mixture upon the steam-bath in a platinum bowl. This precipitate was collected upon a Gooch crucible in which a circular piece of hardened filter-paper was used instead of an asbestos mat. After drying and converting the mass into the black oxide by ignition with an alcohol lamp this material was used directly for analysis. The metal which served as the source of this oxide was known to be free from alkalis and silica (*Proc. Amer. Acad.*, 1897, xxxiii., 120; Chemical News, lxxvii., 20), and as neither the nitric acid nor the ammonia could have introduced either of these impurities, the oxide must have been as pure as possible. It has already been shown that specimens of cobalt prepared by any of the methods described in the preceding parts of this paper are essentially identical (*Proc. Amer. Acad.*, 1897, xxxiii., 120; Chemical News, lxxvii., 20).

A weighed platinum boat, containing several grms. of cobaltic oxide, was placed in the porcelain tube, and, after the air had been exhausted, the portion of the tube containing the boat was heated to full redness. The expelled oxygen was removed with the air-pump; and the tension of the gases in the tube was finally reduced to a small fraction of a millimetre of mercury. The tube was then cooled while the vacuum was still maintained, air was admitted, and the boat was transferred to the weighing bottle and weighed. Successive ignition for periods of about two hours each reduced the weight of the cobaltous oxide slowly by amounts varying from two- to five-tenths of a m.grm., until constant weight within one-tenth of a m.grm. was obtained. During this process the cobaltous oxide, which was light brown at first, gradually assumed a darker tint.

Since complete reduction of this oxide by hydrogen at a temperature which could be borne by hard glass is very slow, the reduction was conducted in a porcelain tube heated by the furnace. The metallic cobalt, which sintered together in such a fashion as to occupy not more than one-fifth the original volume of the oxide, was cooled in a vacuum, and then transferred to the weighing bottle and weighed. Constant weight was obtained without difficulty. (Sintered cobalt does not occlude important amounts of hydrogen, even when cooled in this gas; a paper showing this fact will soon be published).

In the following table are given the results obtained by this method of procedure. The correction to a vacuum standard of +0·000069 grm. per grm. of cobaltous oxide was applied, the specific gravity of the oxide being taken as 5·68 (Joule and Playfair, "Landolt and Börnstein's Tabellen," 1894, p. 134). O = 16·000.

Number of analysis.	Weight of cobaltous oxide in vacuum. Grms.	Weight of cobalt in vacuum. Grms.	Atomic weight of cobalt.
1.	7·04053	5·53779	58·962
2.	6·69104	5·26312	58·974
3.	7·83211	6·15963	58·927
			58·954

From the fact that the above results agree neither among themselves nor with the results obtained from the bromide (58·995), it seemed highly probable that all excess of oxygen had not been removed from the cobaltous oxide. It has been shown that oxides made by ignition of nitrates give off their extra "included" oxygen only upon long continued heating, the gas gradually working its way out by a process of dissociation and re-combination (T. W. Richards, *Proc. Amer. Acad.*, xxxiii., 399; Chemical News, lxxix., 19). Here also the oxygen combined with the innermost parts of the monoxide, too tightly enclosed to escape easily, must have required time to force its way out by the same process.

Accordingly, in the next analysis the oxide was heated for much longer periods in the highest vacuum obtainable. Under these conditions the loss of weight between each two ignitions increased very materially, amounting to more than a m.grm. in each case, and culminated in a loss of 3 m.grms. accompanied by a decided darkening of the oxide. This called to mind an experiment made by Dr. Cushman in his work upon the atomic weight of nickel (*Proc. Amer. Acad.*, xxxiii., 424; Chemical News, lxxvi., 284). In this experiment he ignited nickelous oxide to bright redness in a current of nitrogen. Upon removal from the ignition tube the oxide was found to have been partially converted into metallic nickel, owing doubtless to the dissociation of the oxide into metal and oxygen, and the subsequent carrying away of the oxygen by the current of nitrogen. Cupric oxide, when heated only to 700—800°, is known to dissociate perceptibly into cuprous oxide and oxygen (*Proc. Amer. Acad.*, xxxiii., 421). As a matter of fact, when the cobaltous oxide was removed from the boat, it was found to have been reduced to such an extent that enough metallic cobalt had been formed to alloy with the platinum; thus cobaltous oxide also, at about 800°, dissociates very considerably into cobalt and oxygen. In the zeal to obviate oxidation, the opposite error had been encountered. Obviously the only method of obtaining the monoxide free from the metal was to determine the tension of the oxygen evolved from it at the temperature to which the oxide was heated, and always to keep the tension above this point during the ignition.

In the earlier experiments, an exceedingly slight known leakage of water through the brass stoppers into the tube may explain why the reduction of the monoxide did not occur, for metallic cobalt decomposes water at a high temperature.

Some of the brown monoxide which had suffered partial reduction in the experiment described above, and which consequently must have been free from higher oxides, was now heated in a hard glass tube sealed at one end; and the tube was exhausted to within two-tenths of a millimetre of mercury by means of the Sprengel pump. When the pump was stopped the tension of the gas slowly increased from four-tenths of a millimetre at 400° to one and five tenths m.m. at full redness, the highest temperature obtainable with the Bunsen lamp. The pressure of the

oxygen resulting from the dissociation of the *black* oxide of cobalt was determined to be in the neighbourhood of 630 m.m. at the same temperature. Evidently, if the tension of the oxygen remains below 630 m.m. of mercury, the higher oxides of cobalt cannot exist (except in an imprisoned state); and, on the other hand, if the tension of the oxygen does not fall below one and five tenths m.m. there can be no reduction of the monoxide. In the next analysis the oxygen was removed from the tube as fast as it was evolved until the tension of the gas remaining was about one and five tenths m.m. The pressure inside the tube was kept at this point until heating ceased, when the tube was exhausted as completely as possible. In subsequent ignitions of this boat-load the tube necessarily contained air, not oxygen, and allowance was made for this fact by increasing the pressure five times. In this case also constant weight was obtained only after many ignitions although the differences in the weight of the oxide were small. 7·74242 grms. (in vacuum) of cobaltous oxide yielded 6·09219 grms, of cobalt, corresponding to an atomic weight of 59·068.

Evidently even here traces of metal had been found during the heating of the monoxide. This undesired reduction could have taken place only during the cooling, for at all other times the tension of the oxygen was greater than the dissociation pressure of the monoxide. Measures were then taken to make this error impossible.

On account of the size of both tube and furnace, the cooling was necessarily very slow; this fact is indeed an advantage, because otherwise the life of a tube would be short. Assuming that the tube cooled always at the same rate, the tension of the oxygen from the dissociated oxide after any given interval of time from the cessation of ignition should always be the same. A table embodying such tensions was prepared by repeatedly exhausting the tube to a tension slightly below the supposed point, and allowing the pressure inside the tube to reach a maximum. This table, which was obtained by averaging three different series of observations, is given below. The readings were obtained by means of a McLeod gauge:—

Time from cessation of ignition.	Tension of oxygen resulting from dissociation.
Minutes.	M.m.
15	0·80
30	0·56
45	0·28
60	0·21
75	0·11
90	0·07

These values have no meaning except for the particular process by which they were obtained, but they show plainly how great a chance for error existed when the tube was exhausted completely as soon as ignition ceased.

In the next analysis the procedure was exactly similar to that in analysis 4, except that during cooling the values indicated in the table were maintained. No essential loss of weight took place after the third heating, although several additional ignitions were carried out for the sake of certainty. 10·58678 grms. (in vacuum) of cobaltous oxide yielded 8·32611 grms. of cobalt, corresponding to an atomic weight 58·929.

In the face of this unsatisfactory result, there seemed nothing further to do except to attempt the detection of the presence of impurities. Three possibilities were open: the oxide might contain moisture, enough nitrate might have been carried down by the cobaltous hydrate to cause a small amount of nitrogen and oxygen to be occluded by the oxide, or else some higher oxide of cobalt might be present.

It was easily shown that cobaltous oxide is as a matter of fact slightly hygroscopic. Several grms. exposed for twenty-four hours to an atmosphere saturated with moisture gained 5 m.grms. in weight, most of which was lost in a short time in a desiccator. It was satisfactorily proved, however, by suitable experiments which need not be recounted, that not enough moisture could have crept in during any of the processes used to cause any important error.

In order to prove the absence of included gases, a specimen of oxide which had been treated in the same manner as the material used in analysis 5 was dissolved in hydrochloric acid in an apparatus for measuring the gases evolved during the solution (*Proc. Amer. Acad.*, xxxiii., 403, and *Journ. Chem. Soc. Trans.*, lxxi., 559). No gas was given off; but upon treating the solution with potassic iodide and starch, an unmistakable liberation of iodine was detected. This showed conclusively that some higher oxide of cobalt had remained undecomposed, for pure cobaltous chloride has no immediate effect on hydriodic acid. The presence of the imprisoned higher oxide is undoubtedly the reason for the low value for the atomic weight of cobalt ebtained in this way.

The cobalt itself obtained from the pure oxide by reduction at a high temperature is very permanent in the air, and occludes only traces of hydrogen.

With great reluctance the work upon the monoxide was abandoned at this point; for a substance whose composition varies so widely under conditions which vary so slightly is obviously unfitted for work of the highest accuracy. As in the case of cobaltous bromide, the preparation of this substance even in a reasonably pure state appears to be an impossibility. Unfortunately the accurate estimation of the worst impurity in this case, cobaltic oxide, is a very difficult matter. An attempt to use pure cobaltic oxide itself as the basis of the determination proved to be equally unsatisfactory.

The results of this part of the investigation are nevertheless of considerable import in the light they throw upon previous analyses of the monoxides of both nickel and cobalt as well as upon the accuracy of the results obtained in the analyses of cobaltous halides described in this paper. The material which gave the lowest result contained oxides of cobalt higher than the monoxide, hence these results are too low. On the other hand, the material which gave the highest result was heated under conditions which theoretically should have caused, and evidently did cause, partial reduction of the oxide to metal. The true value of the atomic weight of cobalt must lie between these two extremes, 58·93 and 59·07; and until trustworthy evidence to the contrary has been produced, the most probable value seems to be the mean 58·995, obtained from the analysis of the bromide.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 1st, 1900.

Professor Thorpe, F.R.S., President, in the Chair.

Mr. J. W. Anderson was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Henry Jennings, Marlowes, Hemel Hempstead, Herts; Robert Pettigrew, 50, Cresswell Grove, West Didsbury, Manchester; Geoffrey Surtees Phillpotts, 82, James's Street, Dublin.

Of the following papers, those marked * were read:—

*29. "*On Pilocarpine and the Alkaloids of Jaborandi Leaves.*" By H. A. D. Jowett, D.Sc.

On account of the doubts expressed by Kundsen, Merck, and others, as to the reliability of the work of Hardy and Calmels on this subject, and the recent conflicting statements of Petit and Polonowski and of Merck, the author has undertaken a complete study of these

alkaloids. The present paper deals with the characterisation of pilocarpine, isopilocarpine (pilocarpidine of Petit and Polonowsky), pilocarpidine and jaborine, with a few preliminary experiments on the constitution of the alkaloid. The results, which are given in detail in the paper, may be briefly summarised as follows :—

1. The original papers of Hardy and Calmels are very unsatisfactory, as they contain neither physical constants, nor analyses of the products described. In most cases the author is unable to confirm their result. On the other hand, a satisfactory explanation is afforded of the apparent contradiction between the work of Petit and Polonowsky and that of Merck.

2. The physical constants and description of the salts of pilocarpine, as given by Petit and Polonowski, are generally confirmed, though in several cases there are slight differences, due probably to the use of purer material by the author.

3. The acid character of pilocarpine has been investigated and the previous work of Hardy and Calmels on this subject corrected.

4. The existence of a base, isomeric with pilocarpine, and produced from it by the action of heat or alkali, as previously stated by Petit and Polonowski, is confirmed. As this base is isomeric with and bears a close relation to pilocarpine, it is proposed to call it isopilocarpine, as the term pilocarpidine, previously used for it, must be retained for the product previously described by Harnack under this name. It is shown that isopilocarpine can be distilled unchanged in a vacuum.

5. Some of the physical constants of isopilocarpine and its salts as given by Petit and Polonowski are confirmed, and others corrected.

6. Proof is given of the existence of isopilocarpine in jaborandi leaves, and in the pilocarpine nitrate of commerce.

7. The existence of the pilocarpidine of Harnack and Merck, and their statements regarding its composition, are confirmed. Some of the salts of the base are described.

8. The absence of pilocarpidine in the pilocarpine nitrate of commerce, and in the varieties of jaborandi leaves at present in the market, is proved.

9. The jaborine of commerce is shown to be a mixture of isopilocarpine, pilocarpidine, and a trace of pilocarpine with colouring matter. No evidence has been obtained of the existence of an alkaloid with the properties of jaborine, or of any other than those described in this paper.

10. Several experiments on the constitution of pilocarpine have been repeated, and the results of Hardy and Calmels corrected.

Further experiments on this subject are in progress.

The author has been unable to obtain any evidence confirmatory of the constitutional formula which was proposed by Hardy and Calmels and widely adopted.

Discussion.

Mr. Kingzett referred to his work on pilocarpine, which was communicated to the Society in 1876. He then proposed for this alkaloid the formula $C_{23}H_{35}N_4O_4$, and, although this formula had been modified by later workers to $C_{11}H_{16}N_2O_2$, which was also adopted by Dr. Jowett, he still adhered to the formula he had originally suggested, because it was based on several concordant analytical results.

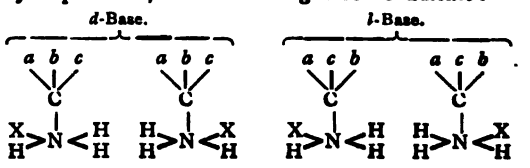
Professor Dunstan said that our knowledge of the chemistry of pilocarpine and the alkaloids of jaborandi was in a very uncertain state, and he was glad to find that Dr. Jowett had commenced its careful revision. The alleged existence of jaborine, an alkaloid antagonistic to pilocarpine, had long been a difficulty in the medicinal employment of this drug. Dr. Jowett appeared to have shown good grounds for the conclusion that no such alkaloid as jaborine exists in the plant, and he had made a serious criticism of the generally accepted results and conclusions of Hardy and Calmels.

*30. "*Isomeric partially Racemic Salts containing Quinquevalent Nitrogen. Hydrindamine Bromocamphorsulphonates, Chlorocamphorsulphonates, and* cis-*π-camphanates.*" By F. Stanley Kipping, Ph.D., D.Sc., F.R.S.

On continuing the investigation of the isomeric hydrindamine bromocamphorsulphonates already briefly described (*Proc.*, 1899, xv., 172), it has been found that both compounds, when decomposed by barium hydroxide, yield a base identical with externally compensated hydrindamine. It is proved that the optical inactivity of the base is not due to racemisation having occurred,—hence the formation of the two bromocamphorsulphonates cannot be attributed to a re-solution of the base into its enantiomorphous components. As to the possibility of the existence of two isomeric forms of bromocamphorsulphonic acid, it is shown that the acid in each salt is the same. Hence it is concluded that the isomeric hydrindamine bromocamphorsulphonates are both partially racemic salts.

The specific rotations of these salts are different in alcoholic solutions of equal concentration ; their specific rotations in 1 per cent aqueous solutions are, for the more sparingly soluble α-salt $[\alpha]_D = +60°$, and for the β-salt $[\alpha]_D = +49{\cdot}7°$, hence the molecular rotations are $[M]_D = +266{\cdot}5°$ and $[M]_D = +220{\cdot}5°$ respectively. As the molecular rotation of bromocamphorsulphonic acid, calculated from the determinations of Walden (*Zeit. Phys. Chem.*, 1894, xv., 196), and from data given in the paper is +270°, the low molecular rotation of the β-salt might be taken to indicate either the existence of an isomeric form of bromocamphorsulphonic acid or the presence of lævorotatory hydrindamine. It is shown that as the salt in question is composed of *d*-hydrindamine α-bromocamphorsulphonate and *l*-hydrindamine α-bromocamphorsulphonate, which salts have probably widely different specific rotations in a non-dissociated condition, the apparently abnormal molecular rotation of the β-salt may be attributed to incomplete ionic dissociation.

The following is the explanation of this form of isomerism suggested by the author ; it is equally applicable to all the other cases of isomerism of this kind which he has observed. When an externally compensated base combines with an optically active acid, four different salts may be produced, as the following formulæ indicate :—



Of these four compounds, no two are mirror images, but as the relation between I. and IV. on the one hand, and II. and III. on the other, would probably be that of mirror images if the group X were not enantiomorphous, it may be presumed that I. and IV. would be formed in equal quantities, and might unite to form a partially racemic salt ; the isomeric partially racemic salt would be similarly produced from II. and III.

Externally compensated hydrindamine unites with chlorocamphorsulphonic acid to form unequal quantities of two salts which are isomeric when anhydrous, but which are usually differently hydrated. The more sparingly soluble α-salt, which is produced in by far the larger quantity, crystallises from water in opaque, very slender prisms, whereas the β-isomeride is usually deposited in more transparent and compact prisms.

The relationship between these two salts is analogous to that of the compounds above described, but the β salt differs from the corresponding salt of the bromo-acid, as it exists in two differently hydrated forms, one of which is indistinguishable in appearance from the α-salt. The conditions which determine the formation of one or other variety of the β-salt are not yet known with certainty.

The α- and β-compounds are both partially racemic salts which are not convertible the one into the other by ordinary solvents, but by repeatedly evaporating a solution of the β-salt with externally-compensated hydrindamine, the α-isomeride is obtained.

The specific rotations of the isomerides in alcoholic solution are practically identical, but when dissolved in water, and especially in chloroform, different values are obtained. Determinations in approximately 1 per cent aqueous solutions gave the following average results:—For the α-salt $[\alpha]_D = +47°$, $[M]_D = +188°$; for the β-salt $[\alpha] = +50{\cdot}5°$, $[M]_D = +202°$.

The molecular rotation of chlorocamphorsulphonic acid was found to be $[M]_D = +189°$, so that the molecular rotations of the partially racemic salts are practically identical with the values they might be expected to give under the above conditions. The two differently hydrated forms of the β-salt were found to have the same specific rotation when calculated for the anhydrous salt.

When externally compensated hydrindamine is neutralised with *cis*-π-camphanic acid, unequal quantities of two isomeric anhydrous salts are obtained. The more sparingly soluble α compound melts at about 192°, the isomeric β-salt at about 172°; when crystallised from water or other solvents, both salts are deposited in very very slender prisms practically identical in appearance. The base liberated from either of these salts by barium hydroxide is optically inactive, and as each salt, when directly decomposed by strong hydrochloric acid, gives optically inactive hydrindamine hydrochloride, it is clear that the two salts do not correspond with the two enantiomorphous hydrindamines, but that both are partially racemic substances.

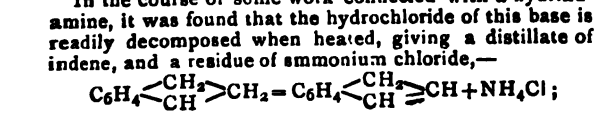
The β-salt may be converted into the α-form by evaporating its aqueous solution with externally compensated hydrindamine, which seems to indicate that the relation between these two salts is similar to that between the isomeric salts of chloro- and bromo-camphorsulphonic acids from which they differ in undergoing hydrolytic dissociation, and this leads to other differences in behaviour, such as the conversion of the one salt into the other in aqueous, and apparently also in alcoholic solution, readering their separation and the determination of their properties a matter of some difficulty.

Determinations of the specific rotations of these salts in solution in methyl alcohol showed wide variations; different preparations of the α-salt, all melting at the same temperature, gave values for $[\alpha]_D$ varying from $-2°$ to $+20°$, and in the case of β-salt from $-6°$ to $-10°$. By repeated fractional crystallisation, these values were considerably raised, the greatest rotation for the α-salt being $[\alpha]_D = +24°$, and for the β-salt $[\alpha]_D = -20°$.

In solution in methyl alcohol, the rotation of the α-salt is very low at first, being $[\alpha]_D = +5°$ a few minutes after solution; after forty-five minutes, it rises to $[\alpha]_D = 24°$, at which it seems to remain constant. The cause of this change is not clear, as the β-salt, in other respects so similar, does not show it. In dilute aqueous solutions, both salts have practically the same rotation, $[\alpha]_D = -16°$, and the molecular rotation calculated from determinations in 1 per cent aqueous solutions is $[M]_D = -53°$ approximately, a value agreeing fairly well with that obtained for *cis*-π-camphanic acid from an examination of its piperidine salt and of its solution in dilute aqueous ammonia.

*31. "*New Syntheses of Indene.*" By F. Stanley Kipping, Ph.D., D.Sc., F.R.S., and Harold Hall, A.I.C.

In the course of some work connected with α-hydrindamine, it was found that the hydrochloride of this base is readily decomposed when heated, giving a distillate of indene, and a residue of ammonium chloride,—



the reaction seems to take place almost quantitatively, and affords the most convenient method yet known for the preparation of this unsaturated hydrocarbon. Trimethylhydrindamine iodide undergoes a similar decomposition when it is heated, yielding indene and trimethylamine hydriodide. The indene obtained by either of these methods is identical with the hydrocarbon isolated from coal-tar by Krämer and Spilker, and also with that obtained by Perkin and Revay by the distillation of hydrindenecarboxylic acid (*Trans.*, 1894, lxvi., 228). The identity of all three preparations was established, the differences between synthetical and coal-tar indene observed by Perkin and Revay (*loc. cit.*) being doubtless due to the presence in the latter of impurities which they were unable to remove.

Trimethylhydrindamine iodide, $C_9H_9N(CH_3)_3I$, is obtained when hydrindamine is treated with methyl iodide at ordinary temperatures; it crystallises well, is dimorphous, and melts at about 190°.

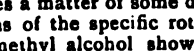
It seems probable that the reactions described above will also occur in the case of the hydrochlorides of other aromatic partially reduced bases, and experiments will be made to test this.

*32 "*Potassium Nitrito-hydroximidosulphates and the Non-existence of Dihydroxylamine Derivatives.*" By E. Divers and T. Haga.

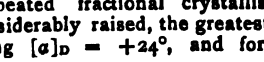
Potassium nitrite forms crystalline compounds with the normal, the 2/3 normal, and the 5/6 normal potassium hydroximidosulphates, all readily decomposed by solution in water, but re-crystallisable from sufficiently concentrated solutions of potassium nitrite or of this salt and some potassium hydroxide. Their formulæ are—

$KNO_2,2K_3NS_2O_7,4OH_2$;
$KNO_2,K_2HNS_2O_7$;
$7KNO_2,2K_5H(NS_2O_7)_2,3OH_2$;
$3KNO_2,K_5H(NS_2O_7)_2,OH_2$;
$7KNO_2,3K_5H(NS_2O_7)_2$.

The discovery of these salts has enabled the authors to destroy what little evidence there was for the existence of *dihydroxylamine* compounds, two of which have been described by Raschig. One of these he treats as being dipotassium monosulphonated dihydroxylamine,—



and the other, more easily obtained, as the anhydro-compound of this with potassium monosulphonated dihydroxylamine,—



The authors have followed Raschig's methods pretty closely and with a sufficient degree of success, and have found his preparations indistinguishable from synthetically prepared compounds, somewhat impure, of nitrite with the 5/6 normal hydroximidosulphate, and the 2/3 normal hydroximidosulphate respectively. According to them, the andydro-'dihydroxylamine' compound was $KNO_2,K_2HNS_2O_7$, whilst the other was impure $3KNO_2,K_5H(NS_2O_7)_2,OH_2$. Two other salts prepared by Raschig, which have figured respectively as an isomeride of Pelouze's hyponitrososulphate and as an isomeride of the 5/6 normal hydroximidosulphate, are considered by the authors to be also only impure nitritohydroximidosulphates.

*33. "*Identification and Constitution of Fremy's 'Sulphazotised Salts of Potassium.'*" By E. Divers and T. Haga.

By treating a solution of potassium nitrite and hydroxide with sulphur dioxide, Fremy in 1845 obtained a series of salts, and by their hydrolysis or oxidation, other salts, all sulphonated derivatives of nitrous acid. The constitution of several of these has remained undetermined; attempts to reproduce some of them have

failed to such an extent as to make even their existence doubtful. The authors have been fairly successful in obtaining Fremy's results, and with the help of the knowledge summarised in the preceding abstract, are able to express the constitution of his salts with confidence. His *sulphazate*, which neither Claus nor Raschig succeeded in obtaining, was made without difficulty by following Fremy's directions. It is the potassium nitrito-2/3-normal hydroximidosulphate partly converted, by treatment with alkali, into the normal hydroximidosulphate. His *sulphasite* is the nitrito-5/6-normal hydroximidosulphate of which the formula is given in the preceding abstract. His *metasulphazate* is described as a jelly, which on pressure in a cloth became wax-like. The authors have obtained a salt in fine, silky fibres in all other respects apparently identical with that of Fremy, and this proved to be potassium nitrito-normal hydroximidosulphate, mixed with a little normal hydroximidosulphate. His *metasulphazotate*, as obtained by the authors, proved to be the potassium nitrito-2/3-normal hydroximidosulphate, mixed or combined with a little normal hydroximidosulphate. The constitution of Fremy's *basic* and *neutral sulphazotates*, *sulphazidate*, *sulphammonate*, and *sulphamidate* have been already determined, whilst his *sulphazilate* and *metasulphazilate* appear to have the formulæ $ON:(SO_3K)_2$ and $ON:(SO_3K)_3$.

(To be continued).

PHYSICAL SOCIETY.

Special Meeting, March 9th, 1900.

Prof. EVERETT, F.R.S., Vice-President, in the Chair.

A PAPER on "*The Damping of Galvanometer Needles*" was read by Mr. M. SOLOMON.

The solution of the equation of motion for a magnetic needle, swinging in a uniform magnetic field, points to the conclusion that the ratio of the period to the logarithmic decrement is independent of either the moment of the needle or the strength of the controlling field, and is simply a function of the damping coefficient, and the moment of inertia of the moving system. This ratio should therefore be constant if these latter quantities are constant. Experiments to test the constancy of period to logarithmic decrement have been conducted at the Central Technical College at various times since 1891, and they have invariably pointed to a variation in the value of the ratio. The object of the present paper is to discover the cause of this variation. It may be due to an alteration in the moment of inertia or to an alteration in the damping coefficient. If the control magnets are either directly above or directly below the needle there is no chance of any change in the moment of inertia. The damping coefficient depends on three things: — (1) viscosity of the air, (2) viscosity of the suspension, and (3) eddy currents. The author has carried out experiments with a galvanometer on open circuit and finds a constant value for the ratio; the viscosity of the air and suspension therefore cause no variation. Upon closing the circuit and repeating the experiments the value of period over logarithmic decrement alters. The variation is therefore due to eddy currents. The damping factor due to eddy currents may vary owing to three causes:— (1) change in moment of needle due to change in field strength, (2) effects of self-induction, (3) effects of rise of temperature on the resistance of the coils. The author points out that the two latter causes would tend to alter the ratio in the wrong direction, and he therefore concludes that the variation is due to an alteration in the strength of the swinging needle, produced by altering the strength of the controlling field.

Mr. BLAKESLEY said it was interesting to note the fact that the ratio of period to decrement was independent of the controlling field. In the case of a condenser discharging this ratio is independent of the capacity, in the case of a tuning fork, of the rigidity and in the case of water oscillating up and down in a U-tube, of the acceleration due to gravity.

Mr. ROSENBAUM said that the ratio considered was constant in the case of a Nalder D'Arsonval galvometer.

Mr. SOLOMON said that his arguments did not apply to galvanometers of this description because the swinging system was not a magnetic needle, but simply a coil.

A paper on "*The Distribution of a Gas in an Electric Field*" was read by Mr. G. W. WALKER.

The author has considered a gas as consisting of a number of molecules, each containing two atoms of equal mass, one positively and the other negatively charged with electricity. When under the action of electrical forces some of the molecules split up, and we arrive eventually at a steady state in which there is a definite number of undissociated molecules and of free positive and free negative atoms. Treating the problem as one-dimensional, the potential at any point is expressed in in general by elliptic functions, and is therefore periodic. Applying the results to the case of a vacuum-tube it is found that there is superimposed upon the gradual fall of potential along the tube, minor periodic variations which it is suggested are connected with the striæ of discharge. Both the matter density and the electric density are periodic along the tube. If the places of maximum matter density coincide with the places of minimum electrical action, then whether luminosity is due to collisions or recombinations there will be maximum luminosity at these points. In general, these points do not coincide, and thus the positions of maximum luminosity are not clearly defined. The analysis leads to the conclusion that the distance between the striæ is inversely proportional to the density of the gas and to the current strength, and these facts have been experimentally verified.

Mr. C. E. S. PHILLIPS exhibited a "*Surface Tension Lecture Experiment.*"

The effects of surface tensions were exhibited by placing water between two pieces of microscope cover glass. When the glasses are circular they set in any position, and one can be made to rotate upon the other. If the plates are square or elliptical they set in a definite position, to which they immediately return if displaced. Mr. Phillips pointed out how two circular discs with liquid between could be used from which to suspend the moving system of a galvanometer.

Mr. COCHRANE suggested the use of some liquid which would evaporate less quickly than water.

Mr. BLAKESLEY asked what accuracy could be obtained with such an arrangement, and what weight it would be possible to support without squeezing out the water.

The meeting then adjourned until March 23rd.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. CXXX., No. 8, February 19, 1900.

Researches on the Isomerism of Sulpho-cyanic Derivatives.—M. Berthelot.—This paper is unsuitable for abstraction.

Method of Preparation of Arsenides, Alkaline Stibnides, and some Alloys of the Alkaline Metals.—P. Lebeau.— Arsenic or either gaseous or liquefied arseniuretted hydrogen react on the alkaline metals, but do not give pure crystallised arsenides. Liquid arseniuretted hydrogen does not dissolve the alkaline

metals either at −80° or at the ordinary temperature and pressure. Arsenium metals comparable with the ammonium metals are not formed. The employment of an alkaline metal as solvent of its arsenide, and the elimination of excess of metal by means of liquefied ammonia gas, constitutes an excellent method of preparing crystalline arsenides. This method admits of general use, and allows of the preparation of definite crystalline alloys of the alkaline metals.

Nitrogen Iodide.—C. Hugot.—The author obtains the bodies $NI_3.3NH_3$, $NI_3.2NH_3$, and $NI_3.NH_3$ in a well crystalline form. These can be considered merely as ammoniacal combinations of nitrogen iodide, on account of the facility with which they lose their ammonia. These substances are prepared by the action of liquid ammonia gas on nitrogen iodide under varying experimental conditions.

Meconine, Opianic Acid, and Hemipinic Acid.—Emile Leroy.—The author determines the heat of formation and combustion of these substances, and obtains the following numbers:—*Meconine*, $C_{10}H_{10}O_4$,—

Heat of combustion at constant volume and pressure	1090·4 cal.
Heat of formation from its elements	+197·7 „

Opianic Acid—

Heat of combustion at constant volume	1260·7 cal.
„ „ „ pressure	1261·0 „
„ formation from its elements	+190·4 „

Hemipinic acid, $C_{10}H_{10}O_6$, an acid resulting from the oxidation of opianic acid.

Heat of combustion at constant volume	1024·9 cal.
„ „ „ pressure	1024·6 „
„ formation from its elements	+263·5 „

Rapid Method of Estimating Carbonic Acid in Various Gases.—Léo Vignon and Louis Meunier.—Into the flask containing carbonic acid mixed with other neutral gases little by little a solution of lime-water of known strength, reddened with phenolphthalein, is introduced. The lime-water only retains its rose colour after complete saturation with carbonic acid. The operation is rendered more rapid and more complete by adding a little ethyl alcohol; this substance facilitates the formation of insoluble calcium carbonate. The proportion of carbonic acid present is measured by the quantity of lime-water used to obtain the red colouration, a deduction being made for the quantity absorbed to allow of the colouration of the phenolphthalein. This method can be used to estimate the CO_2 in furnace gases, &c.

Volumetric Estimation of Boric Acid.—Alfred Stock.—Whilst trying to analyse a certain number of borides, the author employed Jones's method, by which, in an acid solution of boric acid, the mineral acids are neutralised by a mixture of iodide and iodate of potassium, which has no action on the boric acid. The author finds the method not satisfactory, and introduces various improvements:—(1) It is essential to avoid even minute traces of carbonic acid; this end can only be attained by boiling all the reagents to eliminate the last traces of CO_2. (2) Although the metals of the alkalis and alkaline earths do not affect the titration of boric acid, other metals, whose solutions are precipitated by soda, cause interference, and have to be carefully eliminated from the solution before titration of the boric acid.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxi., Nos. 20-21.

Analysis of some Commercial Samples of Carbide of Calcium.—Henri Moissan.—Already inserted.

The Preparation of Crystallised Calcium.—Henri Moissan.—Already inserted.

Preparation and Properties of Hydride of Calcium.—Henri Moissan.—Pure crystallised calcium is placed in a nickel boat in a glass tube traversed by a current of pure dry hydrogen. On raising the temperature of the calcium to a dull red, it takes fire in the atmosphere of hydrogen. The gas is rapidly absorbed, and a white substance is formed, which is hydride of calcium. This hydride is stable at a high temperature, and is an energetic reducing agent; its formula is CaH_2. On contact with water it decomposes with violence. In this compound the hydrogen is comparable with the metalloids (carbon or phosphorus), and not with the metals. There are in reality two series of hydrides, in one of which the hydrogen seems to be in solution in the metals; and the others forming at a more or less elevated temperature, having all the characteristics of definite chemical compounds.

Preparation and Properties of Nitride of Calcium.—Henri Moissan.—When a morsel of pure crystallised calcium is placed in an atmosphere of pure dry nitrogen, no change takes place in the cold, but on raising the temperature there is at first a slow absorption of gas and a change of colour in the metal, which gradually becomes yellow, then bronze. On further increasing the temperature the colour deepens, and at a dull red heat the combination takes place with incandescence. When the reaction takes place at about 1200° the compound obtained is crystalline; it answers to the formula N_2Ca_3. It reacts readily with chlorine, bromine, iodine, oxygen, sulphur, and phosphorus, and is destroyed by carbon at a high temperature. With anhydrous alcohol it gives ammonia and ethylate of calcium. It also decomposes on contact with cold water, giving off ammonia.

The Action of Magnesium on Saline Solutions.—D. Tommasi.—When magnesium is treated with a solution of chloride of potassium, hydrogen is given off, the liquid becomes cloudy, and acquires a slightly alkaline reaction. The product of this reaction is hydrate of magnesium. The chloride of potassium is not decomposed by this reaction, but plays no other part beyond helping the oxidation of the magnesium. The reaction thus only takes place between the water and the magnesium, according to the following equation:—

$$Mg + 2H_2O = Mg(OH)_2 + H_2.$$

The author then gives a list of chlorides and sulphates of a number of metals, and the action their solutions have on metallic magnesium.

Fluorine in the Mineral Waters of Portugal.—A. J. Ferreira da Silva and Alberto d'Aguiar.—Already inserted in full (see vol. lxxxi., p. 4).

MISCELLANEOUS

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 5th instant, His Grace the Duke of Northumberland, K.G., President, in the Chair. Mrs. Colenso, Miss A. C. Colthurst, Professor J. D. Everett, Professor P. F. Frankland, Mr. A. C. Ionides, Mrs. A. B. Kempe, Mr. A. G. Low, Colonel W. T. Makins, Colonel F. Rhodes, Mr. W. M. Still, Major F. Trench-Gascoigne, Dr S. West, Mrs. West, and Miss H. M. White, were elected Members of the Royal Institution.

On the Oxime and the Phenylhydrazone of Xanthone.—C. Graebe and P. Roeder.—Meyer and Spiegler, basing their ideas on the fact that xanthone and euxanthone do not give any products of condensation either with hydroxylamine or phenylhydrazine, came to the conclusion that the compounds of the euxanthone group were lactones. Graebe and Feer have, however, furnished the proof that xanthone ought to be considered

as a cyclic ketone, and its formation by means of phenylsalicylic acid has confirmed this view. In fact, the authors have succeeded in preparing the phenylhydrazone of xanthone by means of its thio-derivative $O<\frac{C_6H_4}{C_6H_4}>CS$, to which they give the name xanthione. This compound was itself prepared from xanthone-phenylimine, obtained by heating a mixture of equal parts of aniline and 2.2'-dioxybenzophenone for two or three hours, at 200°. Xanthone-phenylimine, dissolved in alcohol and saturated with H_2S, gives xanthione which occurs in the form of long needles possessing dichroism to a strong degree, and melting at 156°. Concentrated H_2SO_4 dissolves it with a yellow colour with green phosphorescence. Xanthione, heated in alcoholic solution with hydrochlorate of hydroxylamine and carbonate of sodium, gives the corresponding oxime, the xanthone oxime, $O<\frac{C_6H_4}{C_6H_4}>C=N.OH$, which concentrated sulphuric acid dissolves yellow, with blue phosphorescence, and which melts at 161°. Xanthione, heated for four or five hours in alcoholic solution with excess of phenylhydrazine, gives the phenylhydrazone of xanthone,—

$$O<\frac{C_6H_4}{C_6H_4}>C=N-NH.C_6H_5,$$

which crystallises in alcohol in golden-yellow needles fusible at 152°.—*Berichte*, vol. xxxii., p. 1688.

MEETINGS FOR THE WEEK.

Monday, 19th.—Society of Arts, 8. (Cantor Lectures). "Photography of Colour," by E. Sanger Shepherd.

Tuesday, 20th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

Wednesday, 21st.—Society of Arts, 8. "The Use and Abuse of Food Preservatives," by Dr. Samuel Rideal.

— Microscopical, 7.30. Exhibition of Slides of New, Rare, and Foreign Rotifera, by C. F. Rousselet, Curator R.M.S.

Thursday, 22nd.—Royal Institution, 3. "Equatorial East Africa and Mount Kenya," by H. J. Mackinder, M.A.

Friday, 23rd.—Physical, 5. "An Electro-magnetic Experiment," by Prof. S. P. Thompson, F.R.S. "Some Experiments illustrating Syntony" and "An Electrical Micrometer," by P. E. Shaw.

— Royal Institution, 9. "Some Modern Explosives," by Sir Andrew Noble, K.C.B., F.R.S., M.Inst.C.E.

Saturday, 24th.—Royal Institution, 3. "Polarised Light," by The Rt. Hon. Lord Rayleigh, M.A., D.C.L., &c.

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Lent Term.—Monday, January 8, to Saturday, Apr 7.

Easter Term.—Monday, April 30, to Saturday, July 28.

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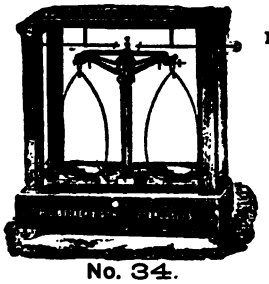

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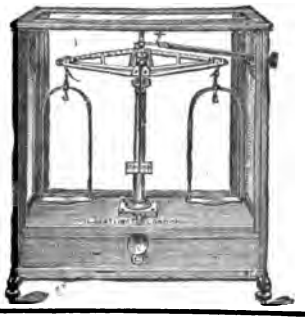

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THE CHEMICAL NEWS.

VOL. LXXXI., No. 2104.

ON THE SPECIFIC HEAT OF METALS AND THE RELATION OF SPECIFIC HEAT TO ATOMIC WEIGHT.*

By W. A. TILDEN, D.Sc., F.R.S.,
Professor of Chemistry in the Royal College of Science, London.

WITH AN APPENDIX BY PROFESSOR JOHN PERRY, F.R.S.

THE experiments described in this paper were begun with the object of assisting in the determination of the relative values of the atomic weights of cobalt and nickel, but were continued with the further purpose of testing the validity of the law of Dulong and Petit.

The metals cobalt and nickel closely resemble each other in density, melting-point, and other physical properties, as well as in atomic weight. The pure metals were prepared for the purposes of these experiments with the most scrupulous care, the cobalt by taking advantage of the slight solubility of purpureo-cobaltamine hydrochloride in strongly acid solutions, and the nickel by deposition from the carbonyl compound and subsequent solution of the metal and electro-deposition. Both were fused by means of an oxyhydrogen flame, and afterwards shaped into bars. For the estimations of specific heat between 15° C. and 100° C. the differential steam calorimeter of Professor Joly was employed.

The mean specific heat of cobalt within these limits of temperature was found to be 0·10303.

The mean specific heat of nickel was found to be 0·10842.

In order further to test the method and the conclusion from the case of cobalt and nickel, gold was compared with platinum, and copper with iron.

The following are the mean specific heats for these pure metals after fusion, and within the same limits of temperature:—

Gold	0·03035
Platinum	0·03147
Copper	0·09232
Iron	0·10983

When these values for the specific heats are multiplied by the respective atomic weights of the several metals, the products are not constant, as the law of Dulong and Petit would seem to require if applicable at all temperatures.

The influence of impurities on the specific heat of several metals was then investigated and a number of results are given, from which it appears that small quantities of carbon or other non-metallic element tend to increase the specific heat appreciably, while the presence of a small quantity of a foreign metal seem to produce little effect.

A series of calorimetric experiments were next made by the method of mixtures on the two pure metals cobalt and nickel, at the temperature of solid carbon dioxide, −78·4°, and at that of boiling oxygen, −182·5°.

The results, which are given below, show that as the temperature is reduced the value for nickel declines more rapidly than that for cobalt, and hence, that when the mean results are plotted out, the curves steadily approach each other.

The mean specific heats of cobalt and nickel now stand as follows:—

Temperature.	Cobalt.	Nickel.
From 100° to 15°	0·10303	0·10842
,, 15° ,, −78·4°	0·0939	0·0975
,, 15° ,, −182·4°	0·0822	0·0838

and by calculation from the last two results from −78·4 to −182·4, 0·0712, 0·0719.

It seems probable that at absolute zero the values of the products of specific heat, multiplied by atomic weight, would be identical, or differ only by the very small amount due to experimental error.

Note added March 3.—Further experiments made, since the date of communication, upon the metals silver, copper, iron, and aluminium, show, however, that this suggestion will not be realised. The mean specific heat of silver between 15° and 182·4°, for example, is 0·0519, while from 100° to 15° it is 0·0558. The decrease of specific heat at the lower temperature is therefore much less than in the case of cobalt and nickel.

APPENDIX.

The following calculations have been made by E. R. Verity, Assoc. R.C.S., and H. L. Mann, Assoc. R.C.S.:—

If cobalt and nickel were in the states of perfect gases, their specific heats at constant volume would be—

$$k_0 = 0{\cdot}04123 \text{ for cobalt.}$$
$$k_0 = 0{\cdot}04145 \text{ for nickel.}$$

This is on the assumption that k_0 multiplied by the atomic weight is the same as for hydrogen. It is not unreasonable to assume that in no state can the substance have a smaller specific heat than k_0. It is easy to show that for any of the metals the specific heat at atmospheric pressure K is not more than 2 per cent different from the specific heat under any other condition, such as great hydrostatic pressure, to keep its volume constant. Our ignorance of the molecular state of a solid is so great, that we cannot even speculate on how it is that when 1 grm. of cobalt (we have much the same figures for other metals) at 50° C., rises in temperature to 51° C., whether it is allowed to expand freely or is subjected to great hydrostatic pressure which prevents expansion, the energy 0·041 enters it as what may be called the real sensible heat, and the energy 0·062 enters it as some kind of energy of disgregation, necessary because of change of temperature, and having nothing to do with change of volume or pressure. The facts are not explainable by assuming that the atomic weights are wrong, because, as we see from cobalt and nickel, K approaches the value k_0 at low temperatures. Indeed, if the following formula is correctly deduced from the above measurements, we may say that in the solid state the product of the atomic weight and specific heat may be anything between what it is for hydrogen and 2·7 times this amount. The formula is—

$$K = k_0 + \frac{bt^2}{1 + ct^2};$$

where t is the absolute temperature.

	Cobalt.	Nickel.
k_0	0·0412	0·0415
b	$1{\cdot}764 \times 10^{-8}$	$1{\cdot}764 \times 10^{-8}$
c	$2{\cdot}55 \times 10^{-7}$	$2{\cdot}35 \times 10^{-7}$

This formula must give fairly correct values of K from −180° C. to 100° C.

Chemical Society.—The Annual General Meeting of the Society, for the election of Officers and other business, will be held on Thursday, March 29th, at 3 o'clock in the afternoon. Presentation of the Longstaff Medal to Professor W. H. Perkin, jun., F.R.S. In the evening the Bunsen Memorial Lecture will be delivered by Sir Henry E. Roscoe, F.R.S. The Chair will be taken at 8.30.

* Bakerian Lecture, read before the Royal Society, March 8, 1900.

HOW TO IGNITE A HYDROGEN JET WITH NO POSSIBILITY OF EXPLODING THE GENERATOR.

By C. G. HOPKINS.

The following are perhaps fair samples of the directions usually found in text-books and manuals for igniting a jet of recently generated hydrogen gas:—

"After the action has continued for a minute or two apply a lighted match" (Remsen, "Introduction to Chemistry," p. 64).

"When all the air is expelled, not less than ten minutes, light the jet of hydrogen" (Newbery, "Laboratory Notebook," eighteenth practice).

"When the gas is coming off freely, light the jet" (Shepard, "Elements of Inorganic Chemistry," p. 38).

As is well known, with any of the above directions, inexperienced students do continue to explode hydrogen generators with annoying frequency, and it is believed that a method of igniting a jet of hydrogen gas with no possible danger of exploding the generator will be welcomed generally by teachers of introductory and qualitative chemistry.

The following method, used by the writer with his classes for a number of years, is absolutely safe, and causes no loss of time whatever:—

As soon as the action begins, collect the escaping gas in a test-tube, and, when it is thought to be full of pure gas, remove 2 or 3 feet from the generator and ignite the hydrogen in the test-tube; then immediately *attempt* to light the jet of hydrogen *with the hydrogen flame contained in the test-tube*. If the gas is explosive it will explode in the test-tube and *leave no flame*. If, on the other hand, a flame remains in the test-tube with which the jet can be ignited, it is certain that the gas in the generator is no longer explosive. Hence, the caution—Never light the hydrogen jet except with the hydrogen flame obtained as just described. The student may try to ignite the jet by this method as often as he wishes until he succeeds, and if the hydrogen is properly generated the jet will be ignited in less than a minute.—*Journal of the American Chemical Society*, xxi., No. 7.

THE PRECIPITATION OF COPPER BY ZINC.*

By JOHN C. SHENGLE and EDGAR F. SMITH.

At various times during the past year attempts have been made in this Laboratory to make a direct comparison between silver and cadmium, with the hope that in this way the atomic mass of the latter metal might be definitely established. The most carefully purified metallic cadmium in weighed amounts was allowed to act upon various soluble salts of silver. The precipitation of the latter metal was rapid and complete. The results, while very fair in a quantitative respect, could not be used for the purpose designed. The precipitated silver contained cadmium. It was not undissolved portions of the latter, but metal originating from the salt which was acted upon, which salt became in some way encased in the silver so that the most painstaking efforts failed to eliminate it. Nor did it seem to make any difference as to which silver salt was acted upon. Cadmium, in small amounts it is true, but nevertheless cadmium, was invariably discovered in the beautiful deposits of silver.

When copper salts were substituted for silver salts, the cadmium threw out the copper quickly and in calculated amounts, but it also was contaminated with varying quantities of cadmium, so that the scheme originally planned for the determination of the atomic mass of cadmium was abandoned.

This negative experience suggested the idea of testing the copper precipitated by metallic zinc. This being a method recommended for the quantitative estimation of copper, we concluded to ascertain if possible whether it carried or retained any zinc. Pure copper sulphate was prepared, and metallic zinc was also obtained pure by the distillation method of Morse and Burton (*Am. Chem. Journ.*, 1888, x., 148).

The determinations were conducted in the usual manner, but instead of weighing the precipitated copper it was dissolved in nitric acid and estimated by means of the electric current. The liquor poured off from this deposit was examined for the zinc.

Analyses.

1. Twenty c.c. of a copper salt solution, containing 0·1739 grm. of copper, was precipitated by metallic zinc. The deposit of copper was filtered, thoroughly washed, dried, and dissolved in acid, and its solution electrolysed, the filtrate from the copper deposit being used for the determination of any contaminating zinc:—

	Copper present. Grm.	Copper found. Grm.	Zinc found. Grm.
I.	0·1739	0·1742	0·0067

In the second and third determinations the results were—

	Copper present. Grm.	Copper found. Grm.	Zinc found. Grm.
II.	0·1739	0·1741	0·0027
III.	0·2609	0·2612	0·0033

Twenty-two precipitations were made with varying conditions. Zinc was found in all of the precipitated copper. As a rule this method gives fair results, but it is notwithstanding interesting to know that the good results must be due largely to a balancing of errors.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, vol. xxi., No. 10.

A NEW TEST FOR COCAINE.

By GEORGE L. SCHAEFER.

There are two well known tests for determining the freedom of commercial cocaine salts from other coca alkaloids. These are the permanganate test for detecting cinnamyl-cocaine, and the ammonia test, popularly known as McLagan's test, for detecting the presence of the coca alkaloids which are resistant to permanganate. While it is generally admitted that the permanganate test is sufficient to detect the presence of cinnamyl compounds, chemists have expressed some doubt regarding the value of McLagan's test. The writer has for some time been conducting experiments with the object of finding a substitute for McLagan's test which would allow of the rapid and accurate determination of the presence in cocaine salts of the coca alkaloids not indicated by the permanganate test.

As the result of numerous determinations, I have devised a test based on the fact that the chromates of these alkaloids are much less soluble than cocaine chromate, both in water and in water acidulated with hydrochloric acid. The relative solubility of the chromates in acidulated water is about 1 to 500 in the case of cocaine chromate, and 1 in 5000 in the case of the residual alkaloidal chromates.

I therefore offer the following as a simple and satisfactory method of determining the purity of cocaine salts:—0·05 grm. cocaine hydrochloride is dissolved in 20 c.c. of distilled water, mixed with 5 c.c. of a 3 per cent solution of chromic acid, and to the mixture 5 c.c. of a 10 per cent solution of hydrochloric acid are added. It is advisable to keep the temperature of the solution at 15° C. If the cocaine hydrochloride be pure a clear solution will result. If other than traces of foreign coca bases be present the solution becomes cloudy at once, or in a few minutes, according to the amount of impurity present.

It is advisable to make the test side by side with a specimen of known purity for comparison.—*Journal of the American Chemical Society*, xxi., No. 7.

ON THE NORMAL PRESENCE AND THE LOCALISATION OF ARSENIC IN THE ORGANS OF ANIMALS.

By A. GAUTIER.

Various circumstances have led me to study the action of arsenical preparations on the animal economy; I have particularly observed that cacodylic acid, $As(CH_3)_2O_2H$, obtained by Bausen in 1843—an acid which has remained unemployed since its discovery—is of remarkable efficacy in certain specific diseases (anæmia, malaria, goitre, phthisis), although all the poisonous and even chemical properties of the arsenic have disappeared in this very soluble body, which, however, contains 54·3 per cent of metalloidic arsenic.

Since my communication to the Academy of Medicine last June, cacodylic acid is being made in France in a state of great purity, and is now sold by hundreds of kilogrammes.

In the course of this research I was led to ask by what mechanism does arsenic act on the economy. Various considerations led me to think that it fixes on the nucleinic matter where it doubtless replaces phosphorus to a certain extent. On the other hand, remembering the fact that in certain diseases of the thyroid gland a general treatment with arsenic and iodine is advantageous; also that arsenic and iodine are often associated in nature, and not forgetting the fact that, in the normal state, iodine exists in a fairly large quantity in the thyroid gland, it appeared to me that it might be possible for arsenic to be one of the constituent principles of this gland, having the same value as the iodine, and perhaps normally present in other tissues of the body.

These ideas have been confirmed by my researches.

I have discovered that arsenic exists normally and in smaller proportions in the thymus and the brain, and as traces in the skin and hair.

The normal existence of arsenic in animal bodies appear to contradict all the teachings of toxicology. Thousands of careful experiments have been made on human beings without detecting arsenic in the organs. These facts are explained, on the one hand, by the imperfect methods generally employed for the destruction of the organic matter (Cl; ClO_3K+HCl; attack by *aqua regia*, and distillation, &c.), methods which, on account of the disengagement of volatile chlorinised fumes, cause the partial or complete loss of the small quantities of arsenic.

It is, on the other hand, explained by the consideration that arsenic, according to the experiments I am about to describe, is absent from most of the human organs. It is only met with in the thyroid, the thymus, the brain, and as traces in the bones and skin.

I do not speak for the organs I have not yet examined.

I have vainly tested for arsenic in 100 to 200 grms. of the following organs:—

The liver of sheep, dogs, pigs, and calves;
The spleen of dogs and oxen;
Loins of pigs;
The pitutary gland of sheep;
The flesh of dogs;
The stomachic mucus;
And pig's blood (250 grms. defibrinated).

I have never found arsenic in these organs, or, if present, it was in a state of such dilution that my method, which enables one hundredth of a milligramme to be detected in 100 grms. of tissue, showed no trace.

Arsenic is, on the other hand, constantly present in the thyroid gland, and although its quantity is small I have since the commencement of this research been able to detect it in only 5·2 grms. of the fresh thryoid gland of the dog.

45 grms. of the fresh thyroid of a pig gave an arsenic ring weighing about 0·3 m.grms.; or 0·67 m.grms. per 100 grms. of the fresh organ, or nearly seven-tenths of a m.grm. per kilo. This would he about 3 m.grm. per kilo. of the dried gland.

100 grms. of the thyroid of sheep gave me an arsenic ring weighing 0.05 grm.; which corresponds to 2 m.grms. of arsenic per kilogrm. of the dried gland.

All these figures are a little low, which has since been explained by a small error in my method.

After having made the correction, I operated on 127 grms. of human thyroid gland taken from six persons who had not taken any metallic medicaments, and, above all, no arsenic during their illness. I obtained a ring of arsenic weighing nearly 1 m.grm. (0·95 m.grm.).

All these experiments were verified by repeating them twice with sheep's liver totally free from arsenic, as a check. It was treated exactly like the thyroid. All these check experiments gave negative results.

Arsenic thus exists normally in the thyroid gland of herbivorous and carnivorous animals. In the case of man, we have found about 1 m.grm. per 127 grms.; that is, 1/127,000th part of the weight of the fresh thyroid. This small quantity suffices for the gland to perform its functions which are of importance, and depend on the constant presence of arsenic in this specific organ, as there is no thyroid without arsenic and no health without the thyroid.

The gland no doubt obtains this important element from the food we take, though in a state of extreme dilution. Stein (*Journ. f. Prakt. Chem.*, li., p. 302; liii., p, 37) has, in fact, recognised its presence in a number of vegetables—rye-straw, potatoes, turnips, cabbages, &c. I have recently found it myself in several foods of animal origin, especially milk.

Arsenic exists in considerable quantity in the thyroid gland, in smaller quantity in the thymus and the brain, and as traces in the skin. In what form does it exist in these organs?

I thought that, seeing the chemical analogies, arsenic ought to be found in the thyroid, especially in the form of arsenical nucleines forming the nucleus of the cells (*kernucleines*), or the basophil granulations of the protoplasma (*paranucleines*). It might also form part of certain lecithines.

To make sure of the existence of arsenic in the nuclein of the thyroid, I separated these nucleines by pepsic digestion.

This, as is known, peptonises the albumenoids of the protoplasm while the nucleines remain in the residue. In this manner I digested 100 grms. of fresh thyroid from sheep with 0·5 grm. of a pepsic liquor. After fifty-four hours at 38°, there remained a magma formed of indigestible materials, among which could be distinguished an iron grey pulverulent matter, which was found to be very much iodised. The peptones formed and the residue were separated by filtration. I satisfied myself that the peptones did not contain a trace of arsenic. The residue was put to digest at 38° with ammonia diluted with 40 times its volume of water, to dissolve the nucleines; it was then filtered, and the solution precipitated with acetic acid.

The nucleines thus separated, which I found to be very rich in iron (2 m.grms. sufficed for estimating this element), were collected on a filter, washed, and dried. 1·21 grms. of these nucleines, weighed in the dried state, the quantity extracted from 100 grms. of moist thyroid, gave a very good ring of arsenic.

There therefore exist in the normal state in the thyroid, and in much smaller proportion in the thymus, the brain, and the skin, arsenical nucleines (*arsenucleines*), which are liable to be split up by acids into *arsenucleic acids* and albumenoids. These arsenucleic acids (and the arsenucleines themselves) behave as ferments, if we apply the observations of Lilienfeld on the ordinary phosphorised leuco-nucleines. Their constant presence in the normal thyroid gland shows us the necessity of arsenical treatment when illness, or the destruction of the gland, in part or totally deprives the economy of the arsenic which is necessary to it.

Then appears more particularly myxœdemia, which specially affects the thyroid, the brain, and the skin; that is to say, the three organs in which I have observed that arsenic always exists in the normal state.

The proof of the normal existence of arsenic in the animal economy is important not only in furnishing the proof, drawn from the constant presence of arsenic in the thyroid, the brain, and the skin, of the definite relations, chemical and functional, which unite these three organs; but it also shows the influence that almost infinitesimal doses of certain active elements have on the functions of animal life. One human thyroid gland contained about 0·17 m.grm. of arsenic; for a man weighing 67 kilogrms. these 17 hundredths of a m.grm., represent 1/400,000,000th of the total mass. This four hundred millionth is enough to enable the gland to operate normally and the health is maintained. The health suffers when the arsenic diminishes.

There is still another point arising from this research, and that is to decide what specific functions, more or less latent, are proceeding in our organs, thanks to the presence of certain specific elements of which the major part are still probably ignored.

Among these elements of which we are already aware, I would mention manganese, a principle of oxidising fermentation according to M. Bertrand; iodine, a specific element functioning in the thyroid; arsenic, which I have just found in the arsenical nucleines; fluorine, which is found in the bone cells. There is therefore still room for research in every organ, and, thanks to the most delicate methods, for the different elements that we might consider likely, by being substituted as above for the chemical analogues, to modify the functioning of the organ by reason of certain special needs, such would be selenium in place of sulphur; negative sulphur substituted for oxygen; copper, zinc, or manganese replacing iron; phosphorus, arsenic, or even vanadium itself playing the part of nitrogen in the more or less complex molecules. This forms a complete new branch of biological chemistry full of promise for the future.

I need hardly mention what would follow these possibilities from the etiological and therapeutical point of view of illnesses.

Finally, toxicologists will have, from the medico legal-expert point of view, to take notice of these observations which have established the normal existence of arsenic in certain organs, as well as its complete absence from the major part of the tissues and the blood of animals.

It is necessary to add, for the benefit of those who wish to repeat the experiments, that the detection of arsenic in the organs should be made, not by the methods too often used, by which the chlorinised fumes carry off and possibly lose the arsenic, but by the much more certain method described by me in 1876 (*Ann. Chim. Phys.*, Series 5, viii., p. 384)—a method which I have further improved during my present research.—*Bull. Soc. Chim.*, Series 3, xxxiii., No. 1.]

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING FEBRUARY 28TH, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, March 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Feb. 1st to Feb. 28th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month has been exceptionally heavy, no less than 4·23 inches having been measured; the average fall for February is 1·76 inches, we therefore have an excess of 2·47 inches; the total excess for the first two months of this year is now 2·61 inches, or 66·5 per cent on the thirty years' average.

Our bacteriological examinations of 492 samples have given the results recorded in the following table; we have also examined 32 other samples, from special standpipes, &c., making 524 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	682
New River, filtered (mean of 116 samples) ..	18
Thames, unfiltered (mean of 24 samples) ..	8086
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 270 samples)..	66
Ditto ditto .. .. highest	992
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 24 samples) ..	692
River Lea, from the East London Company's clear-water wells (mean of 34 samples) ..	13

The exceptionally high rainfall of 4·23 inches during the month, following a long drought at this time of the year, has resulted in the organic carbon being higher, and the bacteriological purity of some of the filtered waters being lower, than the average of the last few months. This has been especially the case in three of the Thames-derived waters, the filter wells of which on some occasions have given abnormally high numbers. So far as we can ascertain, the working of the filters has been conducted with all the customary care and precautions. Similar abnormal results have usually followed the breaking up of a long drought. On careful examination it is satisfactory to know that the organisms in these exceptional samples are harmless river microbes, and we have been unable to detect among them any other kind of organism. As on previous occasions, the filter beds that gave these abnormal samples of water were again in a few days working satisfactorily.

It will be observed that, in spite of the floods, the bacterial quality of the New River and the Lea supply has been exceptionally good, and amongst the 150 samples of these waters examined no abnormal results were found.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

The Contaminated Waters of the Wells of Guillotière and Brotteaux à Lyon.—H. Causse.—The waters of the wells of Guillotière and Brotteaux have undoubtedly been the cause of typhoid fever. The author, therefore, has examined the waters, and perceived the bacillus *Bacterium termo* to be present in large quantities. He found no albumenoids in the waters, nor nucleo-proteids. —*Comptes Rendus*, cxxx., No. 9.

LIQUID HYDROGEN.*

By Professor DEWAR, M.A., LL.D., F.R.S., &c.

From the year 1878, when the experiments of Cailletet and Pictet were attracting the attention of the scientific world, it became a common habit in text-books to speak of all the permanent gases, without any qualification, as having been liquefied, whereas these experimentalists, by the production of an instantaneous mist in a glass tube of small bore, or a transitory liquid jet in a gas expanding under high compression into air, had only adduced evidence that sooner or later the static liquid form of all the known gases would be attained. Neither Pictet nor Cailletet in their experiments ever succeeded in collecting any of the permanent gases in that liquid form for scientific examination. Yet we meet continually in scientific literature with expressions which lead one to believe that they did. For instance, the following extract from the *Proceedings of the Royal Society*, 1878, illustrates this point very well:—"This award (Davy Medal) is made to these distinguished men (Cailletet and Pictet) for having independently and contemporaneously liquefied the whole of the gases hitherto called permanent." Many other quotations of the same kind may be made. As a matter of fact six years elapsed, during which active investigation in this department was being prosecuted, before Wroblewski and Olszewski succeeded in obtaining oxygen as a static liquid, and to collect liquid hydrogen, which is a much more difficult problem, has taken just twenty years from the date of the Pictet and Cailletet experiments.

Wroblewski made the first conclusive experiment on the liquefaction of hydrogen in January, 1884. He found that the gas cooled in a capillary glass tube to the boiling point of oxygen, and expanded quickly from 100 to 1 atmosphere, showed the same appearance of sudden ebullition lasting for a fraction of a second, as Cailletet had seen in his early oxygen experiments. No sooner had the announcement been made, than Olszewski confirmed the result by expanding hydrogen from 190 atmospheres, previously cooled to the temperature given by liquid oxygen and nitrogen evaporating under diminished pressure. Olszewski, however, declared in 1884 that he saw colourless drops, and by partial expansion to 40 atmospheres, the liquid hydrogen was seen by him running down the tube. Wroblewski could not confirm Olszewski's results, his hydrogen being always obtained in the form of what he called a "liquide dynamique," or the appearance of an instantaneous froth. Olszewski himself seven years later repeated his experiments of 1884 on a larger scale, confirming Wroblewski's results, thereby proving that the so-called liquid hydrogen of the earlier experiments must have been due to some impurity. The following extract from Wroblewski's paper states very clearly the results of his work on Hydrogen:—

"L'hydrogène soumis à la pression de 180 atm. jusqu'à 190 atm., refroidi par l'azote bouillant dans la vide (à la température de sa solidification) et détendu brusquement sous la pression atmosphérique présente une mousse bien visible. De la couleur grise de cette mousse, où l'œil ne peut distinguer des gouttelettes incolores, on ne peut pas encore deviner quelle apparence aurait l'hydrogène à l'état de liquide statique et l'on est encore moins autorisé à préciser s'il a ou non une apparence métallique. J'ai pu placer dans cette mousse ma pile thermo-électrique, et j'ai obtenu suivant les pressions employées des températures de −208° jusqu'à −211° C. Je ne peux pas encore dire dans quelle relation se trouvent ces nombres avec la température réelle de la mousse ou avec la tempêrature d'ébullition de l'hydrogène sous la pression atmosphérique puisque ja n'ai pas encore la certitude que la faible durée de ce phénomène air permis à la pile de se refroidir complètement. Néanmoins, je crois aujourd'hui de mon devoir de publier ces résultats, afin de préciser l'état actuel de la question de la liquéfaction de l'hydrogène."—*Comptes Rendus*, 1885, c., 981.

It is well to note that the lowest thermo-electric temperature recorded by Wroblewski during the adiabatic expansion of the hydrogen (namely, −211°) is really equivalent to a much lower temperature on the gas-thermometer scale. The most probable value is −230°, and this must be regarded as the highest temperature of the liquid state, or the critical point of hydrogen, according to his observations. In a posthumous paper of Wroblewski's on "The Compression of Hydrogen," published in 1889, an account appears of further attempts which he had made to liquefy hydrogen. The gas compressed to 110 atmospheres, was cooled by means of liquid nitrogen under exhaustion to −213·8°. By suddenly reducing the pressure, as low a temperature as −223° on his scale was recorded, but without any signs of liquefaction. This expansion gives a theoretical temperature of about 15° absolute in the gas particles. The above methods having failed to produce static hydrogen, Wroblewski suggested that the result might be attained by the use of hydrogen gas as a cooling agent. From this time until his death in the year 1888, Wroblewski devoted his time to a laborious research on the isothermals of hydrogen at low temperatures. The data thus arrived at enabled him, by the use of Van der Waal's formulæ, to calculate the critical constants, and also the boiling point of liquid hydrogen.

Olszewski returned to the subject in 1891, repeating and correcting his old experiments of 1884, which Wroblewski had failed to confirm, using now a glass tube 7 m.m. in diameter instead of one of 2 m.m. as in the early trials. He says: "On repeating my former experiments, I had no hope of obtaining a lower temperature by means of any cooling agent, but I hoped that the expansion of hydrogen would be more efficacious, on account of the larger scale on which the experiments were made." The results of these experiments Olszewski describes as follows: "The phenomenon of hydrogen ebullition, which was then observed, was much more marked and much longer than during my former investigations in the same direction. But even then I could not perceive any meniscus of liquid hydrogen." Further, "*The reason for which it has not hitherto been possible to liquefy hydrogen in a static state, is that there exists no gas having a density between those of hydrogen and of nitrogen, and which might be for instance* 7—10 (H=1). Such a gas could be liquefied by means of liquid oxygen or air, as cooling agent, and be afterwards used as a frigorific menstruum in the liquefaction of hydrogen."

Professor Olszewski, in 1895, determined the temperature reached in the momentary adiabatic expansion of hydrogen at low temperatures, just as Wroblewski had done in 1885, only he employed a platinum-resistance thermometer instead of a thermo-junction. For this purpose he used a small steel bottle of 20 or 30 c.c. capacity, containing a platinum-resistance thermometer; in this way, the temperatures registered were regarded as those of the critical and boiling points of liquid hydrogen, a substance which could not be seen under the circumstances and was only assumed to exist for a second or two during the expansion of the gaseous hydrogen in the small steel bottle.

The results arrived at by Wroblewski and Olszewski are given in the following table, and it will be shown later on that Wroblewski's constants are nearest the truth.

	Wroblewski, 1885.	Olszewski, 1895.
Critical temperature.. ..	−240°	−234°
Boiling point	−250°	−243°
Critical pressure..	13 atm.	20 atm.

The accuracy of Wroblewski's deductions regarding the chief constants of liquid hydrogen following from a study of the isothermals of the gas is a signal triumph for the

* A Lecture delivered at the Royal Institution, January 20, 1899.

theory of Van der Waals, and a monument to the genius of the Cracow physicist. From these results we may safely infer that, supposing a gas is hereafter discovered in small quantity four times more volatile than liquid hydrogen, having a boiling point of about 5° absolute, and therefore incapable of direct liquefaction by the use of liquid hydrogen, yet by a study of its isothermals we shall succeed in finding out its most important liquid constants, although the isolation of the real liquid may for the time be impossible.

In a paper published in the *Philosophical Magazine*, September, 1884, "On the Liquefaction of Oxygen and the Critical Volumes of Fluids," the suggestion was made that the critical pressure of hydrogen was wrong, and that instead of being 99 atmospheres (as deduced by Sarrau from Amagat's isothermals) the gas had probably an abnormally low value for this constant. This view was substantially confirmed by Wroblewski finding the critical pressure of 13·3 atmospheres, or about one-fourth of that of oxygen. The CHEMICAL NEWS, September 7, 1894, contains an account of the stage the author's hydrogen experiments had reached at that date. The object was to collect liquid hydrogen at its boiling point, in an open vacuum vessel, which is a much more difficult problem than seeing it in a glass tube under pressure and at a higher temperature. In order to raise the critical point of hydrogen to about −210°, from 2 to 5 per cent of nitrogen or air was mixed with it. *This is simply making an artificial gas containing a large proportion of hydrogen which is capable of liquefaction by the use of liquid air.* The results are summed up in the following extract from the paper:—"One thing can, however, be proved by the use of the gaseous mixture of hydrogen and nitrogen, namely that by subjecting it to a high compression at a temperature of −200° and expanding the resulting liquid into air, a much lower temperature than anything that has been recorded up to the present time can be reached. This is proved by the fact that such a mixed gas gives, under the conditions, a paste or jelly of solid nitrogen, evidently giving off hydrogen, because the gas coming off burns fiercely. Even when hydrogen containing only some 2 to 5 per cent of air is similarly treated, the result is a white solid matter (solid air) along with a clear liquid of low density, which is so exceedingly volatile that no known device for collecting it has been successful." This was in all probability the first liquid hydrogen obtained, and the method is applicable to other difficultly liquefiable gases.

Continuing the investigations during the winter of 1894, and the greater part of 1895, the author read a paper before the Chemical Society in December of that year, entitled: "The Liquefaction of Air and Research at Low Temperatures" (*Proc. Chem. Soc.*, 1895, No. 158), in which occasion was taken to describe for the first time the mode of production and use of a Liquid Hydrogen Jet. At the same meeting Professor William Ramsay made an announcement of a sensational character, which amounted to stating that my hydrogen results had been not only anticipated but bettered. The statement made to the

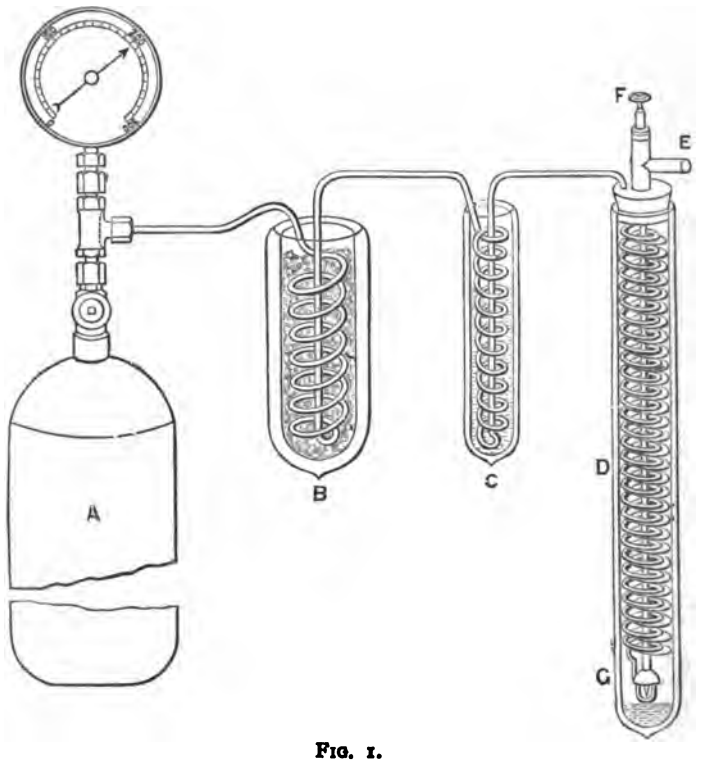


FIG. 1.

Society by Professor Ramsay, reads as follows:—"*Professor Olszewski had succeeded in liquefying hydrogen, and from unpublished information received from Cracow, he (Ramsay) was able to state that a fair amount of liquid had been obtained, not as a froth, but in a state of quiet ebullition, by surrounding a tube containing compressed hydrogen by another tube also containing compressed hydrogen at the temperature of oxygen boiling at a very low pressure. On allowing the hydrogen in the middle jacket suddenly to expand, the hydrogen in the innermost tube liquefied, and was seen to have a meniscus. Its critical point and its boiling point, under atmospheric pressure, were determined by means of a resistance thermometer.*"—*Proc. Chem. Soc.*, 1897—1898, No. 195.

This announcement of Professor Ramsay's had from its very specific and detailed experimental character the merit of the appearance of being genuine, although it was never substantiated, either by the production of the Cracow document, or by any subsequent publication of such important

results by Professor Olszewski himself. My observation at the time on Professor Ramsay's communication was that quotations had been made in my paper from the most recent publications of Professor Olszewski in which he made *no mention of getting "Static Hydrogen or of seeing a meniscus" or of getting as Professor Ramsay alleged "a fair amount of liquid, not as a froth, but in a state of quiet ebullition."* To achieve such a result would require a very different scale of experiment from anything Professor Olszewski had so far described. Naturally an early corroboration of the startling statement made by Professor Ramsay as to this alleged anticipation was expected, but strange to say Professor Olszewski published no confirmation of the experiments detailed by Professor Ramsay in scientific journals of date immediately preceding my paper or during the following years, 1896, 1897, or up to May, 1898. The moment the announcement was made by me to the Royal Society in May, 1898, that, after years of labour, hydrogen had at last been obtained as a static liquid, Professor Ramsay repeated the story to the Royal Society that Olszewski had anticipated my results (basing the assertion solely on the contents of the old letter received some two and a half years before), in spite of the fact that during the interval he, Professor Ramsay, must have known that Professor Olszewski had never corroborated in any publication either the form of the experiments he had so minutely described, or the results which were said to follow. Challenged by me at the Royal Society Meeting on May 12, 1898, to produce Olszewski's letter of 1895, he did not do so, but at the next meeting of the Society, Professor Ramsay read a letter he had received during the interval from Professor Olszewski, denying that he had ever stated that he had succeeded in producing static liquid hydrogen. This oral communication of the contents of the new Olszewski letter (of which it is to be regretted there is no record in the published proceedings of the Royal Society) is the only kind of retraction Professor Ramsay has thought fit to make of his published mis-statements of fact. No satisfactory explanation has yet been given by Professor Ramsay of his twice-repeated categorical statements made before scientific bodies of the results of experiments which, in fact, had never been made by their alleged author. The publicity that has been given to this controversy makes it imperative that the matter should not be passed over, but once for all recorded.

The report of a Friday Evening Discourse on "New Researches on Liquid Air" (*Proc. Roy. Inst.*, 1896), contains a drawing of the apparatus employed for the production of a jet of hydrogen containing visible liquid. This is reproduced in Fig. 1. A represents one of the hydrogen cliners; B and C, vacuum vessels containing carbonic acid under exhaustion, and liquid air respectively; D is the coil, G the pin-hole nozzle, and F the valve. By means of this hydrogen jet, liquid air can be quickly transformed into a hard solid. It was shown that such a jet could be used to cool bodies below the temperature that it is possible to reach by the use of liquid air, but all attempts to collect the liquid hydrogen from the jet in vacuum vessels failed. No other investigator improved on my results (*Proc. Chem. Soc.*, 1895, No. 158), or has indeed touched the subject during the last three years. The type of apparatus used in these experiments worked well, so it was resolved to construct a much larger liquid-air plant, and to combine with it circuits and arrangements for the liquefaction of hydrogen. This apparatus took a year to build, and many months have been occupied in the testing and preliminary trials. The many failures and defeats need not be detailed.

(To be continued).

Determination of Sulphur in Sulphites.—A. Bourgougnon (*J. Am. Chem. Soc.*, xx., 469).—The sulphite in dilute hydrochloric acid solution is treated with a solution of hydrogen peroxide, and the sulphuric acid formed is precipitated and weighed as barium sulphate.

A REVISION OF THE ATOMIC WEIGHT OF COBALT.*

Third Paper.—The Analysis of Cobaltous Chloride and Oxide.

By THEODORE WILLIAM RICHARDS and GREGORY PAUL BAXTER.

(Concluded from p. 127).

Critical Review of Earlier Work.

In the light of the information gained during the protracted investigation which has just been described, it is possible to discover the reasons for many of the unusual discrepancies in the older work upon the atomic weight of cobalt. Following is a chronological list of this work, with references to the original articles. The atomic weight of oxygen, upon which the appended values are based, is taken as 16·000.

1826	Rothhoff (*Pogg. Ann.*, viii., 184)	59
1857	Schneider (*Pogg. Ann.*, ci., 387)	60·0
1858	Marignac (*Bibl. Univ. de Geneve*, i., 372) ..	58·8
1860	Dumas (*Ann. Chem. Pharm.*, cxiii., 25) ..	59·1
1863	Russell (*Journ. Chem. Soc.*, [2]. i., 51) ..	58·7
1866	Sommaruga (*Sits. Wien. Akad.*, liv., [2], 50)	60·
1867	Winkler (*Zeit. Anal. Chem.*, vi., 18)	59·4
1867	Russell (*Journ. Chem. Soc.*, [2], vii., 294)..	59·1?
1868	Weselsky (*Ber. d. d. Chem. Gesell.*, ii., 592)	59·1
1871	Lee (*Am. Journ. Sci. and Arts*, [3], ii., 44)	59·2
1886	Zimmermann (*Ann. der Chem.*, ccxxxii., 324)	58·9
1889	Krüss and Schmidt (*Ber. d. d. Chem. Gesell.*, xxii., 11)	—
1889	Winkler (*Ber. d. d. Chem. Gesell.*, xxii., 891)	—
1891	Remmler (*Zeit. Anorg. Chem.*, ii., 221) ..	58·8
1892	Schützenberger (*Comptes Rendus*, cxiv., 1149)	60·1
1893	Winkler (*Zeit. Anorg. Chem.*, iv., 10).. ..	59·8
1894	Winkler (*Zeit. Anorg. Chem.*, iv., 462) ..	59·5
1895	Winkler (*Zeit, Anorg. Chem.*, viii., 1).. ..	—
1895	Winkler (*Zeit. Anorg. Chem.*, viii., 291) ..	—
1896	Hempel and Thiele (*Zeit, Anorg. Chem.*, xi., 73)	58·9
1897	Richards and Baxter (*Zeit. Anorg. Chem.*, xvi., 362)	59·0
1898	Winkler (*Zeit. Anorg. Chem.*, xvii., 236) ..	—
1899	Richards and Baxter (*Proc. Amer. Acad.*, xxxiv., 351)	59·0
1899	Richards and Baxter (the present paper) ..	59·0

All except four of these investigations were accompanied by similar ones upon the atomic weight of nickel, and in general the criticisms made by Richards and Cushman in a recent paper upon this subject may be applied unchanged to the present argument. It is only necessary to call attention here to the features in which differences existed.

The favourite method for the determination of both constants has been the reduction of the oxides. It is evident from our work upon this method that Russell's cobaltous oxide (1863) must have been contaminated with a higher oxide; that Zimmermann's material (1886), treated with greater care in more perfect apparatus, was more nearly normal, although still impure; and that Schützenberger (1892) must have driven off some of the oxygen, which should have been weighed, by his employment of a high temperature. It was reserved for Hempel and Thiele (1895) to discover by varying the conditions of work that cobaltous oxide was incapable of giving constant results, although they did not attempt to find the reason of the inconstancy. Their irregular observations were really a proof of the thoroughness and care of their work, while the wonderful constancy of Zimmermann's results simply shows a constancy of imperfect conditions.

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxv., No. 3, 1899.

The far greater dissociation tension of the higher oxide of *nickel* makes the method much less dangerous in this case; hence the similar results for the atomic weight of nickel are both more concordant and more nearly correct than those for the atomic weight of cobalt.

The four investigations which had no parallel in the case of nickel were those of Sommaruga, Weselsky, Remmler, and Hempel and Thiele. The first of these need not claim serious attention, for there can be no doubt that the result (60·0) is far too high. Weselsky's contribution was very similar to Lee's, which has been treated in Richards and Cushman's criticism. Since the work was obviously crude and incomplete, we must ascribe its accurate average result (58·96) to a compensation of errors.

Two more investigations remain to be considered. In 1891, when the "Gnomium" dispute was at its height, Remmler entered the fray. He fractionated cobaltous hydrate by treating a large quantity of this precipitate with strong ammonia. Each fraction was subjected to a protracted purification, which, although effective in removing heavy metals, introduced more impurity than it removed. His final solution of cobaltous nitrate, from which he formed the oxide by ignition, contained the alkali and silica taken up by at least six successive evaporations to dryness in glass or porcelain, in three of which the solution was ammoniacal. With such hopelessly contaminated material, it is no wonder that the results from twenty-four fractions varied between limits which were nearly 1 per cent apart. As his methods of igniting the monoxide to constant weight in an inert gas was subject to the same errors which have been discussed in the case of Russell and Zimmermann, no further comment is necessary. The average of 24 determinations was 58·80.

Hempel and Thiele, in 1895, at first attempted to find the ratio CoO : Co from analyses of the monoxide. This part of their research has been already discussed.

The remainder of their work consisted in the quantitative preparation and analysis of cobaltous chloride. They were unable to prepare it in a wholly anhydrous condition, and realised this fact; hence the part of their calculation depending upon the weight of the chloride was unsatisfactory. The final step of their investigation was the weighing of the argentic chloride obtainable from the chloride combined with a known weight of cobalt. Although not wholly satisfactory, this part of their research far excelled the earlier work of a similar kind (Dumas) and yielded one of the most trustworthy values for the atomic weight of cobalt (59·91 to 59·94) which had been obtained at that time.

It is clear, from a careful consideration of the details just referred to, that all the wide variations in the older results are explicable. Hence no serious argument exists against the atomic weight of cobalt (58·995, if O = 16) determined by our analyses of cobaltous bromide. Since, moreover, the still more recent results which the chloride (less than 59·05) and oxide (58·93 to 59·07) confirm this value, one may assume that it is not greatly in error. In any case, the Harvard analyses of the bromides of nickel and cobalt must stand and fall together, for they were made by methods essentially similar; and they must at least give the *ratio* of the atomic weights (58·706 : 58·995) with some approach to accuracy. Each of these numbers has found quite independent support for its actual as well as the relative value, so that for the present the study of these atomic weights will not be pursued further in this Laboratory.

Glycerides of the Monobasic Acids in C_{2n}.—L. T. C. Scheij.—This paper is a summing up of the densities, molecular volumes, and indices of refraction of water, and glycerin, and butyric, caproïc, caprylic, capric, lauric, myristic, palmitic, and stearic acids, as well as of the triglycerides derived from these acids.—*Revue Trav. Ch. P.-B.*, xviii., p. 169.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 1st, 1900.

Professor Thorpe, F.R.S., President, in the Chair.

(Concluded from p. 130).

*34. "*Some Acids obtained from α-Dibromocamphor.*" By Arthur Lapworth and Edgar M. Chapman.

Camphonic acid is the name the authors propose for the acid obtained by hydrolysing α-monobromocampholid (compare Forster, *Trans.*, 1896, lxix., 51). It has the formula $C_{10}H_{16}O_3$, crystallises from ethyl acetate in prisms melting at 194°, and appears to be dimorphous. It has all the properties of a ketonic acid, a fact which shows conclusively that α-monobromocampholid contains the group—

$$-CBr-\ldots\ldots CO\cdot O-$$

The *oxime*, $CO_2H\cdot C_9H_{15}:N\cdot OH$, forms slender needles melting at 125—127°; the *semicarbazone*,—

$$CO_2H\cdot C_9H_{15}:N_2H\cdot CO\cdot NH_2,$$

crystallises in flattened needles which melt and decompose at 222—223°. The *phenylhydrazone* and *p-bromophenylhydrazone* were obtained as colourless pulverulent substances, which yielded well characterised sodium salts.

Dibromocamphonic acid, $C_9H_{15}OBr_2\cdot CO_2H$, is obtained on adding bromine to a solution of camphonic acid in chloroform; it melts and decomposes at 144°. *Monobromocamphonolactone*,—

$$C_9H_{12}OBr\cdot CO\cdot O,$$

is formed when the sodium salt of the dibromo-acid is boiled with water; it crystallises in glistening needles or prisms melting at 110—111°. *Tribromocamphonolactone*,—

$$C_9H_{11}OBr_3\cdot CO\cdot O,$$

produced by gently warming dibromocamphonic acid with excess of bromine, melts at 166—167°.

The formation of a tribromolactone from camphonic acid, taken in conjunction with the rest of the evidence relating to the derivatives of camphorenic acid, shows, with some degree of probability, that bromocamphorenic acid has the formula—

$$CH\leqslant\begin{matrix}CH_2\cdot CMe_2\\CBr-CH_2\end{matrix}>CMe\cdot CO_2H,$$

and not—

$$CH\leqslant\begin{matrix}CBr\cdot CMe_2\\CH_2-CH_2\end{matrix}>CMe\cdot CO_2H,$$

for which a preference has been expressed by one of the authors (*Trans.*, 1899, lxxv., 1139).

Camphonic acid unites with hydrocyanic acid, and on treating the product with hydrochloric acid, two new *acids*,—

$$C_9H_{15}<\begin{matrix}CO_2H\\CO\cdot O-\end{matrix}\quad\text{and}\quad C_9H_{15}(OH)<\begin{matrix}CO_2H\\CO_2H\end{matrix},$$

are obtained. The former crystallises in long needles melting at 144—145°; the latter forms orthorhombic plates melting, with formation of an anhydride, at 207°, and cannot be obtained by hydrolysing the lactonic acid.

Homocamphoronic acid is rapidly attacked by bromine at 140°, but the product is nearly free from bromine, and is probably a mixture of lactonic acids. *Trimethyl homocamphoronate*, $C_7H_{13}(CO_2Me)_3$, was obtained on treating the acid with phosphorus pentabromide and bromine, and subsequently pouring into methyl alcohol; it boils at 305—308° under atmospheric pressure. *β-Anhydrohomocamphoronic acid*,—

$$C_7H_{13}(CO_2H)<\begin{matrix}CO\\CO\end{matrix}>O,$$

was obtained on allowing anhydrohomocamphoronic chloride to absorb moisture; it crystallises in well-formed plates or prisms melting at 128—129°; on treatment with aniline or *p*-toluidine, it affords an anilinic and a *p*-toluidinic acid identical with the substances obtained from the ordinary anhydro-acid. Camphononic acid could not be made to unite with hydrocyanic acid, remaining unaltered after several months' contact with the anhydrous acid. The *oxime* of camphononic acid, $CO_2H{\cdot}C_8H_{13}{:}N{\cdot}OH$, forms prisms melting at 178—180°; when warmed with sulphuric acid, it appears to undergo structural change, but the product affords camphoronic acid on oxidation.

By adding bromine to a solution of camphononic acid in chloroform, a *compound*, $C_8H_6Br_4$, is obtained, which crystallises in lustrous needles or prisms melting at 77—78°. It is probably a tetrabromoxylene.

*35. "*Spectrographic Studies in Tautomerism. The Absorption Curves of the Ethyl Esters of Dibenzoylsuccinic Acid.*" By W. N. Hartley, F.R.S., and J. J. Dobbie, M.A., D.Sc.

Knorr (*Annalen*, 1896, ccxciii., 70) describes three varieties of the ethyl ester of dibenzoyl succinic acid. Two of these show exactly similar chemical reactions, while they differ widely from one another in their solubility, melting-points, and other physical properties. Knorr regards these bodies as stereoisomeric modifications of the "keto" form of the ester. The third ester differs from the two already referred to in its chemical as well as in its physical properties, and is regarded by Knorr as one of the three possible stereoisomeric modifications of the "enol" ester, or possibly as a mixture of the three modifications. His conclusions, which are based on purely chemical grounds, have been confirmed by the spectrographic examination.

Stereoisomerides, so far as they have been investigated, have shown no differences in the curves of molecular vibrations drawn from their absorption spectra, whereas substances having different constitutional formulæ have invariably different spectra. On applying this test to the ethyl esters of dibenzoylsuccinic acid, it was found that the "enol" ester gave a totally distinct spectrum from the "keto" esters, whilst the spectra of the "keto" esters were indistinguishable from one another. The amount of general absorption caused by the "enol" ester is greater than that produced by the "keto" esters, and while all the esters exhibit selective absorption, the absorption band of the "enol" ester occupies a totally different position from the band of "keto" esters, and is much less persistent.

The result of this investigation exemplifies the value of the spectrographic method, and shows how it might be applied with advantage to the investigation of similar cases of isomerism, either as a guide to the chemical investigation or to confirm the conclusions drawn therefrom, especially when any doubt arises as to whether the isomerides have a different constitution, or merely differ in the arrangement of the atoms in space.

*36. "*The Curves of Molecular Vibrations of Benz-antialdoxime and Benz-synaldoxime.*" By W. N. Hartley, F.R.S., and J. J. Dobbie, M.A., D.Sc.

Benz-antialdoxime and benz-synaldoxime give identical curves of molecular vibrations. Ethereal solutions of the two substances were examined. A layer of either solution 25 m.m. thick, containing 1 m.grm.-molecule in 100 c.c. of pure absolute ether, cut off all rays beyond $1/\lambda$ 3323 (λ=3009).

In a layer 1 m.m. thick of a solution containing 1 m.grm. molecule in 500 c.c. of ether, the spectrum is continuous to $1/\lambda$ 3638 (λ=2748), an absorption band making its appearance between $1/\lambda$ 3638 (λ=2748) and $1/\lambda$ 4321 (λ=2314), which is still discernible in a layer 1 m.m. thick of a solution containing 1 m.grm.-molecule in 2500 c.c. of ether.

37. "*On Campholytic and Isolauronolic Acids.*" By James Walker and William Cormack.

A concentrated solution of potassium ortho-methyl camphorate yields methyl isolauronolate on electrolysis, and the purified isolauronolic acid obtained from this substance is optically inactive. The isolauronolic acid prepared from the campholytic acid of electrolysis is also without optical activity.

When campholytic acid is treated with fuming hydrobromic acid at the ordinary temperature, it speedily solidifies, forming the hydrobromide $C_8H_{14}Br.CO_2H$, which melts with decomposition at about 100°, and is identical with the substance obtained by Noyes. Isolauronolic acid is attacked much more slowly by fuming hydrobromic acid, but eventually forms the same hydrobromide as campholytic acid. This hydrobromide, on treatment with alkali, decomposes according to the equations,—

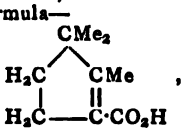
$$\underset{\text{Hydrobromide of campholytic acid.}}{C_8H_{14}Br{\cdot}CO_2H} + 2NaOH = \underset{\text{Sodium campholytate.}}{C_8H_{13}{\cdot}CO_2Na} + NaBr + 2H_2O.$$

$$C_8H_{14}Br{\cdot}CO_2H + 2NaOH = \underset{\text{Isolaurolene.}}{C_8H_{14}} + NaBr + NaHCO_3 + H_2O.$$

These reactions serve to show that campholytic acid is an $\alpha\beta$-unsaturated acid, and that the bromo-acid melting at about 100° is its true hydrobromide. By means of them, isolauronolic acid may be transformed into campholytic acid, which, together with the isolaurolene obtained at the same time is optically inactive.

When a more dilute hydrobromic acid is allowed to remain in contact with isolauronolic acid for a long time, a bromo-acid, $C_8H_{14}Br{\cdot}CO_2H$, is obtained which melts at 132° without decomposition. This is the true hydrobromide of isolauronolic acid, for on treatment with alkali this acid is regenerated from it.

The authors consider campholytic and isolauronolic acids to be stereoisomeric, their structure being best represented by the formula—



derived from the Perkin-Bouveault formula for camphoric acid.

38. "*The Configuration of the Camphoric Acids.*" By James Walker and J. K. Wood.

l-Isocamphoric acid, prepared from *d*-camphoric acid by Marsh's method, has the dissociation constant K=0·00160 at 25°. When boiled with alcohol and a small quantity of sulphuric acid, it yields *ortho-ethyl-l-isocamphorate*, m. p. 73·5°, K=0·00065, $[\alpha]_D = -46{\cdot}8$ for 10 per cent solution in absolute alcohol. *Diethyl l-isocamphorate* is most easily prepared by heating dipotassium *l*-isocamphorate with excess of ethyl bromide in presence of ethyl alcohol for five hours at 100°, when a theoretical yield is obtained. It is an oil for which $[\alpha]_D = -50{\cdot}4°$ for 10 per cent solution in absolute alcohol. *Allo-ethyl-l-isocamphorate* is prepared by the half-saponification of the diethyl salt with the calculated quantity of alcoholic potash. It is a very viscous liquid, having $[\alpha]_D = -22{\cdot}9°$ for 10 per cent solution in alcohol.

It is generally agreed that the molecule of camphoric acid contains two dissimilar asymmetric carbon atoms, which are links in a closed chain, and have attached to them the ortho- and allo-carboxyl groups respectively. On electrolysis of potassium allo-ethyl *d*-camphorate, an ethereal product is obtained, the lowest boiling portions of which have only the allo-carboxyl group and the allo-asymmetric carbon atom, the ortho-asymmetric carbon atom having lost its asymmetry by the removal of the ortho-carboxyl group. This lowest boiling fraction has a strong positive rotation, namely $[\alpha]_D = +29{\cdot}8°$, which must be due to the allo-symmetric carbon atom. If now the allo-asymmetric carbon atom of *l*-isocamphoric acid has the opposite configuration to that of *d*-camphoric acid, the rotation of the corresponding fraction from the

ethereal product of electrolysis of potassium allo-ethyl *l*-isocamphorate should be of the same dimensions but of opposite sign,—that is, it should be strongly negative The rotation of the substance actually produced is, however, strongly positive, namely, $[\alpha]_D = +20{\cdot}9°$, although the original substance and the impurities in the low boiling fraction have a large negative rotation. It appears, therefore, that the allo-asymmetric carbon atoms in *d*-camphoric acid and *l*-isocamphhoric acid have the same configuration, and that the ortho-asymmetric carbon atoms have opposite configurations.

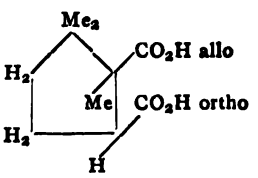
Assuming the configuration RR′ for ordinary camphoric acid, we can represent the configuration of all the camphoric acids by the following scheme:—

	d-Camphoric.	*l*-Camphoric.	*d*-Iso-camphoric.	*l*-Iso-camphoric.
Ortho..	R	L	R	L
Allo ..	R′	L′	L′	R′

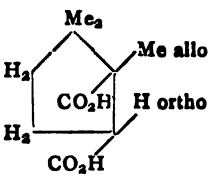
39. "*The Constitution of Camphoric Acid.*" By James Walker.

The Perkin-Bouveault formula for camphoric acid is considered by the author as the most satisfactory hitherto proposed. It represents camphoric acid as a derivative of succinic acid, and thereby accounts in a simple manner for the nature of the unsaturated compounds obtained by electrolysing the sodium alkyl salts of camphoric acid. In particular, the formulæ derived from it for campholytic and isolauronolic acids represent these substances as $\alpha\beta$-unsaturated acids without an asymmetric carbon atom, in accordance with their chemical behaviour and optical inactivity. Perkin's second formula and Bredt's formula, which represent camphoric acid as a derivative of glutaric acid, not only fail to explain the electrolytic production of campholytic and isolauronolic acids, but represent these acids as having an asymmetric carbon atom, and therefore essentially active. Analogy and experiment alike exclude the assumption of a possible racemism.

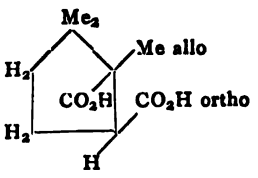
The configuration of the camphoric acids on the basis of the Perkin-Bouveault formula is as follows:—



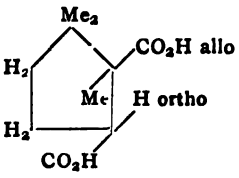
d-Camphoric.



l-Camphoric.



d-Isocamphoric.



l-Isocamphoric.

The dissociation constant of camphoric acid, which seems to be very small for a derivative of succinic acid or glutaric acid, is shown to be only apparently abnormal. The rise in the constant of succinic and glutaric acids when hydrogen is replaced by alkyl, is due to the approximation of the carboxyl groups, and is not the primary effect of the substitution. If the molecule acquires rigidity by ring formation or multiple linking, this approximation is prevented, and the primary effect of the substitution is observed, which is in general a diminution of the constant, as in monocarboxylic acids. It is therefore to be expected that the dissociation constant of camphoric acid, which has one tertiary and one secondary carboxyl, should be less than that of succinic or glutaric acids, which have two primary carboxyls. The very small values of the monocarboxylic acids derived from camphoric acid confirm this view.

40. "*On the Presence of Invertase in Plants of the Gramineæ.*" (I.). By James O'Sullivan.

The difficulty of extracting invertase from the living cells of plants which were proved to contain it was the first point considered. This was overcome in the following manner:—Portions of the roots, culms, and leaves of wheat, oats, and maize plants at various stages of growth, after being well washed with chloroform water, were digested in the cold, and at 49—50°, for different lengths of time, with a cane-sugar solution saturated with chloroform, and the amounts of cane-sugar hydrolysed were determined by the cupric oxide reducing power.

The amounts of cane-sugar hydrolysed are expressed in percentages of the weight of the portions of the plant with which it was digested, and these are taken as expressions for the invertase in those portions.

Check experiments made under similar conditions, (*a*) with cane-sugar solution saturated with chloroform, and (*b*) with chloroform water and portions of the same plants, showed that practically no cane-sugar is hydrolysed, and that the cupric oxide reducing power of the extract is a negligible quantity.

That organisms are not the cause of the hydrolysis of the cane-sugar was proved by "catching up" all the suspended matter in the chloroform water used in washing the portions of the plants, either by means of aluminium hydrate or paper pulp. The aluminium hydrate deposit and the paper pulp, which would contain all the suspended matter—and consequently any organisms present—when digested with similar cane-sugar solutions as in the experiments, gave no more cupric oxide reducing power than the cane-sugar solution and chloroform alone.

41. "*Iodonium Compounds of the Type* $IR^IR^{II}R^{III}$ *and the Configuration of the Iodine Atom.*" By F. Stanley Kipping, Ph.D., D.Sc., F.R.S., and Harold Peters.

It has been shown by Hartmann and Victor Meyer (*Ber.*, 1894, xxvii., 502, 1592) that diphenyliodonium hydroxide, $(C_6H_5)_2I{\cdot}OH$, is obtained when a mixture of iodosobenzene and iodoxybenzene is shaken with silver oxide and water; the authors have made use of this reaction for the preparation of iodonium bases containing different aromatic radicles.

Phenylparatolyliodonium hydroxide,—

$$\begin{matrix} C_6H_5 \\ C_6H_4Me \end{matrix}\!\!>\!I{\cdot}OH,$$

is formed, together with its iodate, from a mixture of iodosobenzene and paraiodoxytoluene,—

$$C_6H_5IO + C_6H_4Me{\cdot}IO_2 + AgOH = \begin{matrix} C_6H_5 \\ C_6H_4Me \end{matrix}\!\!>\!I{\cdot}OH + AgIO_3,$$

and is easily isolated in the form of its *iodide*,—

$$\begin{matrix} C_6H_5 \\ C_6H_4Me \end{matrix}\!\!>\!I{\cdot}I;$$

this salt crystallises from boiling water in small, almost colourless needles, and decomposes at about 153°.

A phenylparatolyliodonium iodide, identical in ordinary properties with the salt just mentioned, is obtained when a mixture of iodosoparatoluene and iodoxybenzene is treated with silver oxide and the product isolated as before.

The identity of these two preparations, if confirmed by further investigation, may be taken as showing that two of the three valencies of the iodine atom are symmetrically situated with respect to the third.

Attempts to resolve the phenylparatolyliodonium base into enantiomorphous components have so far been unsuccessful. When its iodide is treated with silver bromocamphor sulphonate in alcoholic solution, it is converted into *phenylparatolyliodonium bromocamphorsulphonate*,—

$$\begin{matrix} C_6H_5 \\ C_6H_4Me \end{matrix}\!\!>\!I{\cdot}SO_3{\cdot}C_{10}H_{14}OBr,$$

a compound which separates from aqueous alcohol in highly lustrous, well-defined crystals, which contain water of crystallisation, and have in consequence an indefinite melting point.

The same salt is obtained from the two iodides which have been prepared by the different methods stated above.

When this phenylparatolyliodonium bromocamphorsulphonate is fractionally crystallised, the various deposits appear to be identical in ordinary and in optical properties; owing to the slight solubility of the salt in cold water, its specific rotation had to be determined in weak aqueous methyl alcohol, three different fractions giving $[\alpha]_D = +45°$, $+45°$, and $+47°$ respectively.

The samples of salt used contained about 3·6 per cent of water, so that the molecular rotation of the anhydrous substance would be approximately $[M]_D = +283°$; other experiments showed that the presence of methyl alcohol has little effect on the specific rotation, so that, since the molecular rotation of the acid is $[M]_D = +270°$ in aqueous solution, it may be inferred that the iodonium base is optically inactive in all three fractions; unless, therefore, the bromocamphorsulphonate is a partially racemic salt, it may be provisionally concluded that the three iodine valencies, to which the several radicles are attached, are arranged in one plane.

The further investigation of the various points raised in this preliminary note is in progress.

42. "*Note on the Decomposition of Semicarbazones.*" By F. Stanley Kipping, Ph.D., D.Sc., F.R.S.

In working with various semicarbazones—as, for example, with those of dimethylketohexamethylene, ketotetrahydronaphthalene, ketophenoheptamethylene—it was noticed that a brisk evolution of gas usually occurred when such compounds were melted, but that little if any charring took place. It seemed, therefore, that the decomposition products might be easily isolated, and that their examination would show the nature of the change which had occurred.

The only reference in the literature relating to this subject seemed to be that of Scholtz (*Ber.*, 1896, xxix., 610), who, in attempting to prepare pyridazine derivatives, discovered that the semicarbazones of methylethyl ketone, and of other fatty ketones of low molecular weight, were decomposed on dry distillation into ketazines and hydrazodicarbonamide.

The author finds that a similar change occurs in the case of the semicarbazones of aromatic compounds; when benzaldehyde semicarbazone, for example, is heated at a temperature above its melting point, it gives an almost theoretical yield of the *azine*, $C_6H_5{\cdot}CH{:}N{\cdot}N{:}CH{\cdot}C_6H_5$, the nature of which was established by direct comparison with the product of the interaction of benzaldehyde and hydrazine.

This formation of azines by the decomposition of semicarbazones seems also to take place in aqueous solution; when, for example, benzylparatolyl ketone is heated on the water-bath with an aqueous alcoholic solution of semicarbazide hydrochloride and sodium acetate, it seems to be converted in the first place into a colourless semicarbazone in the usual manner, but on continued heating the latter gradually changes into a canary-yellow product which is doubtless the *azine*,—

$$\begin{matrix} C_6H_5{\cdot}CH_2 \\ CH_3{\cdot}C_6H_4 \end{matrix}\!\!>\!C{:}N{\cdot}N{:}C\!<\!\!\begin{matrix} CH_2{\cdot}C_6H_5 \\ C_6H_4{\cdot}CH_3 \end{matrix};$$

the latter is readily soluble in chloroform, but only very sparingly so in hot alcohol, and separates from a mixture of these solvents in yellow needles melting at 172–173°.

It may be concluded from these experiments that the formation of azines from aromatic semicarbazones is also a fairly general reaction.

Preparation of Glycol.—L. Henry.—The author has examined the action of the caustic alkalis on diacetine. The best results were obtained with slaked lime, $Ca(OH)_2$; the lime (1 molecule) is added gradually to the diacetine, and the semi-solid mass which is formed is distilled in vacuo. In this manner 93 per cent of the theoretical return of glycol is obtained.—*R. Trav. Ch. P.-B.*, xviii., p. 221.

CORRESPONDENCE.

A PLEA FOR UNIFORMITY IN ARTIFICIAL MANURE ANALYSIS.

To the Editor of the Chemical News.

Sir.—The strange differences in the results of analysis of manures, as performed by the works chemists and those of the buyers, and between those again and the reference chemists, are a source of never ending annoyance to the chemical manure manufacturers.

What is the cause of these differences? Is it want of experience or care on the part of one or the other chemist? That may be so in some cases, but it is certain that these discrepancies are almost entirely due to the want of uniformity in the methods employed for the analyses.

For instance, the methods used for the estimation of the water-soluble phosphoric acid in the superphosphates are both numerous and very varied; and at the outset there is a great diversity of opinion as to the correct method for extracting it. The following modes of procedure are adopted by different chemists:—

(*a*) 1 grm. superphosphate dissolved by washing on the filter with cold water. (*b*) 1 grm. by boiling for ten minutes or half an hour, and finally washing with boiling water. (*c*) 1 grm. stirred or shaken for half an hour with cold water, decanted, boiled with a new portion of water, and finally washed with boiling water.

In these cases the whole filtrate is tested straight away, whilst in the following only aliquot parts are taken for testing, after the solutions have been filtered:—(*d*) 10 grms. dissolved in 500 c.c. cold water by shaking for half an hour in a shaking apparatus. (*e*) 20 grms. in 500 c.c. cold water for two hours, or over night; and finally (*f*) 2·5 grms. with 1000 c.c. water, &c.

Now the tests themselves are done in varying ways; there are:—(1) The molybdic acid method in different modifications; (2) the uranium acetate method; and (3) the citrate method with different modifications again as to the strength and the quantities to use.

I am not going to criticise these different methods as to whether they are more or less exact or not, as very often it resolves itself into a matter of personal opinion as to the correct method to pursue, and besides that, be they correct or not, the essential point is that they are actually in use, and that they show important differences in the results. I have often observed those over 2 per cent of tribasic phosphate of lime.

About the differences due to the various methods of extraction, I beg to mention that they are less marked or even nothing in fresh and well dissolved supers and in bone manures than in old stock, and in those manures much inclining to revert by lying, such as supers made from Tennessee phosphate.

Partly dissociation of the monobasic phosphate of lime, when dissolved in water—principally if the super is short in acid—into dibasic phosphate and free phosphoric acid, also a varying percentage of Al_2O_3 and Fe_2O_3, may, to a certain extent, explain why some classes of manure are much more sensibly delicate as regards the method of extraction than others.

The following consideration may show how unreasonable and unpractical the use of divergent methods is:—

The value of a delivery of superphosphate is determined by two factors, the weight and the strength, the latter as found by chemical analysis, the unity for the weight being "1 ton," and for the strength "1 per cent tribasic phosphate of lime ($Ca_3[PO_4]_2$) soluble in water." But as different methods for testing the soluble phosphate may give in the *same* superphosphate different results, *i.e.*, may show a different number of units—and it is a fact, the differences are of distinct value for each two methods and each sample, and not accidental—those units arrived at by the different methods, say, for instance. "1 per cent tribasic phosphate of lime soluble in water (by cold

extraction)," and "1 per cent tribasic phosphate of lime soluble in water (by hot extraction)," cannot be of equivalent value, but must also be different. Therefore, using different methods for the same sample, means giving the strength (commercial value), of a superphosphate in different units; and if that is done by the works, buyers, and reference chemist it is in my opinion just as reasonable as if the three interested parties check the weight by a totally different standard; say, for instance, the works send out a delivery of, say, 10 tons (English), the buyer checks the delivery by metrical tons, and as those two would naturally get different results, a referee is asked to decide. But this one uses American (short) tons for weighing the questionable lot. Who could say that is just and reasonable?

In my opinion it is of the greatest interest to chemical manure manufacturers to seek for uniformity of the methods between themselves and the public chemists, and to lay down in this way for the strength of the manure a uniform standard, as already exists for the weight. Because when that is done the manufacturer will not run the risk of being found by the referee under the guarantee with his manures, and of damaging his commercial reputation thereby, although he makes them high enough to show 1 per cent or more over guarantee according to the test of his works chemist.

That has been recognised by the manufacturers of other countries, for instance of Germany, as their methods as published in the small pamphlet entitled "Methoden zur Untersuchung der Kuenstlichen Düngemittel — Verein Deutscher Düngerfabrikanten." (R. Gaertner's "Verlagsbuchhandlung H. Heyfelder," Berlin, S.W., Schönebergerstr. 26), agree in all essential points with those of the official "Versuchs und Controlstationen."

In one word then, "Uniformity of methods is the first condition for uniformity of the results."—I am, &c.,

W. Jerwitz, Ph.D.

31, Nottingham Street, off North Strand, Dublin.

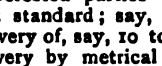
CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 9, February 26, 1900.

Volume Composition of Hydrofluoric Acid.—Henri Moissan.—In a previous paper the author shows that hydrogen and fluorine combine in darkness without the application of any external energy. Hydrofluoric acid is formed with evolution of 38·6 cals. of heat. This method of formation of hydrofluoric acid is, however, too violent for good measurements to be taken, so another method was adopted by which a known volume of fluorine acts on water, and the volume of oxygen produced is measured, $H_2O+F_2=2HF+O$. The oxygen so produced was found to be strongly ozonised. As a result of a series of experiments, it was found that one volume of fluorine unites with an equal volume of hydrogen to give double the volume of gaseous hydrofluoric acid.

Use of Ferricyanides for Oxidation by means of Dehydrogenation. Oxidation of Camphor.—A. Etard.—Ferrocyanides and ferricyanides form, as is well-known, numbers of characteristic precipitates with the alkaloids and other substances containing nitrogen, the action of primitive addition being evident in these cases. The example used for the author's experiments is camphor, and from his results he arrives at the following conclusions:—(1) The ferricyanides are the specific oxidisers; (2) they act more especially on nitrated bodies by dehydrogenation; (3) there are no strong and weak agents of oxidation, but the points of combination and of decomposition depend on the relative structure of the oxidising agent and the oxidised body.

Anhydrous Dimercurammonium Iodide, Amorphous and Crystalline.—Maurice François.—Weyl is said to have obtained anhydrous dimercurammonium iodide in a very unstable form by treating mercuric iodide with liquefied ammonium under pressure. Up to the present the dimercurammonium iodide obtained by the action of ammonia on mercuric iodide and on mercurdiammonium iodide was considered to be hydrated, the formula $Hg_2NI.H_2O$ being given to this substance, or



may be given as the formula, in which HgOH is considered as a monatomic group. The investigation of this substance has led the author to believe that it is always anhydrous; the formula being Hg_2NI and the salt excessively stable. The preparation is effected by means of concentrated ammonia acting on mercurdiammonium iodide or on mercuric iodide, if the latter is pure and anhydrous and a sufficient volume of ammonia is used. It is quicker, however, to prepare the salt by the action of concentrated solutions of potash or soda on mercurdiammonium iodide—an action limited and reversible, like the preceding one.

Estimation of Ammonia and Nitrogen.—A. Villiers and E. Dumesnil.—The inaccuracy of acidimetric estimations in the presence of ammonia salts is well known. To estimate nitrogen the method of transformation into ammonia ought to give better results than the volume estimation of Dumas. The authors find, however, that it is often less exact, and they suggest a simple modification by which the weight of the ammonia or of the nitrogen may be determined with absolute accuracy. The volumetric estimation of ammonia is replaced by a gravimetric determination in the form of ammonium chlorhydrate. Even though the method seems to be longer, since it necessitates an evaparation and a desiccation, it is in reality a more practical and simple method than the volumetric one, since, when the desiccation is finished, there is but a single weighing, and the extra time of the estimation is largely compensated for by the perfect exactness of the results.

Chemical Equilibrium of a System in which Four Gaseous Bodies are present.—H. Pelabon.—This paper is unsuitable for abstraction.

MEETINGS FOR THE WEEK.

Monday, 26th.—Society of Arts, 8. (Cantor Lectures). "Photography of Colour," by E. Sanger Shepherd.

Tuesday, 27th.—Royal Institution, 3. "The Structure and Classification of Fishes," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

Wednesday, 28th.—Society of Arts, 8. "Leather for Bookbinding," by Douglas Cockerell.

Thursday, 29th.—Royal Institution, 3. "Equatorial East Africa and Mount Kenya," by H. J. Mackinder, M.A.

—— Chemical, 3. Annual General Meeting. At 8.30, "Bunsen Memorial Lecture," by Sir Henry E. Roscoe, F.R.S.

—— Society of Arts, 4.30. "The Cultivation, Manufacture, and Use of Indigo—Position of the Industry in India," by Christopher Rawson, F.I.C.

Friday, 30th.—Royal Institution, 9. "Facts of Inheritance," by J. A. Thomson, M.A.

Saturday, 31st.—Royal Institution, 3. "Polarised Light," by The Rt. Hon. Lord Rayleigh, M.A., D.C.L., &c.

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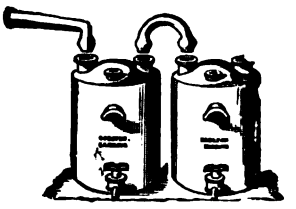

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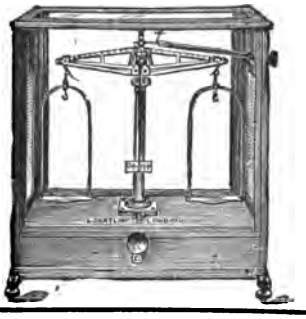

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THE CHEMICAL NEWS.

Vol. LXXXI., No. 2107.

ACTINIUM: A NEW RADIO-ACTIVE ELEMENT.

By A. DEBIERNE.

I showed in a previous paper that there exists in residues from pitchblende, apart from the radium and polonium discovered by M. and Mdme. Curie, a new radio-active substance belonging to the iron group. I further showed that this substance was precipitated by the principal reagents for titanium.

I have continued my researches with the object of obtaining this body, and I have proved that titanium does not resemble it in all its reactions. In addition to the reactions common to all the elements of the iron group (precipitation by ammonia, ammonium sulphide, soda, sodium carbonate, &c.), the following methods appear to be the best to concentrate the new substance:—

1. *Precipitation of the Boiling Solution, slightly Acidulated with* HCl, *by excess of Sodium Hyposulphite.*—The radio-active property is found to reside almost entirely in the precipitate.

2. *Action of Hydrofluoric Acid and Potassium Fluoride on the Freshly-precipitated Hydrates suspended in Water.*—The soluble portion is only slightly active. This method is useful for the separation of titanium from the new substance.

3. *Precipitation of the Neutral Nitrate Solutions by* H_2O_2.—The precipitate contains the radio-active body.

4. *Precipitation of the Insoluble Sulphates.*—Each time that an insoluble sulphate (*e.g.*, barium sulphate) is precipitated in a solution containing this body, the latter is retained, the precipitate being strongly radio-active. This process, which I have found most useful, is entirely different from methods usually employed for the separation of elements.

Oxalic acid, which precipitates the radio-active material well when it is mixed with a tolerably large proportion of the rare earths, seems to be much less efficacious when a number of elements unprecipitated by oxalic acid are present.

These different reactions cannot yet be considered as belonging definitely to the new radio-active substance, because up to the present it has not been obtained sufficiently concentrated. I believe rather that these reactions should be looked upon as the result of retention, analogous to that of iron oxide by barium sulphate.

By using, in a methodical manner, the different methods described above, I was able to extract from the residues of pitchblende the greater part of this new substance which the mineral contained.

The chemical reactions of the most active substance which I obtained, together with the spectroscopic examination, kindly undertaken by M. Demarçay, show that it chiefly consists of thorium. I cannot, however, be sure that it resembles thorium in all its reactions.

If we add a salt of barium or of bismuth to a solution of this substance we can very easily, by the action of ammonia, or H_2S, eliminate the substance having the radio-active property, thereby proving it to be neither radium nor polonium.

I therefore suppose that it is due to the presence of a new radio-active element which I shall call actinium.

It is possible to produce, with the rays emitted by actinium, the same phenomena (fluorescence of barium platinocyanide, photographic impressions, ionisation of gases) as those of the rays emitted by radium and polonium.

Similarly I was able to repeat with this body the experiments MM. Giesel, Meyer, and V. Schweidler and Becquerel on the action of a magnetic field. Under the influence of an intense magnetic field the rays of actinium, or rather a portion of the rays, are deviated, and will impress a photographic plate situated below a lead cell containing the substance.

The deviation is in the same direction as that of the rays of radium or of cathode rays, *i.e.*, it corresponds to a negative charge of the rays.

Finally, actinium, only very feebly, provokes the induced permanent radio-activity discovered by M. and Mdme. Curie on bodies placed in contact or near other radio-active substances. We know that thorium compounds are slightly radio-active. We know further, from what has been said, that actinium seems to be an element allied to thorium. We can therefore suppose that the radio-active property observed in thorium compounds does not belong to this element, but is due to a foreign material. Recent experiments on the radio-activity of thorium made by Rutherford lead also to the same supposition. I propose therefore to try whether it be possible, by utilising the reactions described above, to deprive the thorium compounds of their radio-active property, or to extract from its compounds a substance identical with the actinium extracted from pitchblende.—*Comptes Rendus*, cxxx., No. 14, April 2, 1900.

A NEW METHOD OF SEPARATION OF THE GADOLINITE EARTHS AND PREPARATION OF PURE YTTRIA.*

By W. MUTHMANN and R. BÖHM.

The neutral chromates of the rare earths are slightly more difficulty soluble than the corresponding sulphates; if a neutral solution of any salt of the cerite or gadolinite earths is treated with potassium chromate solution, precipitates are obtained (even at great dilution), which after washing free from alkali correspond, when crystalline, to the general formula, $R_2(CrO_4)_3+nH_2O$. The water content varies with the temperature and the nature of the metal; the chromates mostly crystallise with 8 molecules of water. There certainly exist also, as in the case of the sulphates, other hydrates, which have not yet been examined more closely.

These neutral chromates of the earths afford, as we have discovered, an excellent means for separating earth mixtures; we have been able in comparatively little time, by fractional precipitation with potassium chromate, to accomplish separations which were only attained by formerly used methods with great expenditure of time and material. From this follows the remarkable fact, as we shall presently show, that in fractionating the end is attained most quickly by treating the easily soluble dichromates with yellow potassium chromate. Our experiments are applicable both to the cerium and yttrium earths; the work on the latter group shall now be detailed. When an earth mixture is to be fractionated by precipitation which consists, like the commercial so-called yttria material, of six or more components, a number of rules should be applied in the manipulation, which, though they may appear irrelevant, influence in great measure the success of the separation. In our case—we will now describe our most successful fractionation of the dichromate with potassium chromate—numerous test-experiments indicated the following conditions for the quickest possible separation:—

* *Berichte Deutsch. Chem. Gesell.*, Part 33, No. 1.

1. The solution both of the earth salt and of the potassium chromate must be very dilute.

2. The liquid, during the whole precipitation, must be kept boiling rapidly.

3. The resulting precipitate must be finely divided, and brought into the most intimate possible contact with the liquid.

In order to fulfil these conditions we have adopted the following plan in our experiments:—The liquids for precipitation were heated nearly to boiling in large tubular retorts, and then a stream of steam was led in; to avoid spirting, the necks of the retorts were sloped upwards. Besides the steam delivery tube, there was a second tube, fitted with a stop-cock, which allowed the potassium chromate solution to drop in slowly from a calibrated glass flask. This tube was so arranged that the small amount of precipitation caused by each drop was immediately distributed throughout the whole liquid, which was kept in violent motion. In order to work under similar conditions the liquid was always brought to the original volume by evaporation after each fraction. Our first experiments were carried out in such a way that the chromate solution dropped into the boiling liquid, kept in motion by a stirrer; the effect was also tried at each experiment of pouring in all at once the whole of the chromate solution, reckoned for the single fraction, and of finely dividing the precipitate by boiling and stirring; but the result showed that in neither case was the separation so successful as in the method first described. The time saved is lost again afterwards by the necessity of more frequent repetitions of the precipitation.

The dropping is especially necessary, as otherwise the chromates precipitated are mostly amorphous, and are not crystallisable even after several hours' boiling.

We have now applied the above described method to the most heterogeneous earth mixtures, and in this connection will describe a few test experiments, which afforded us a means of obtaining the purest yttria in relatively short time. We started with an earth mixture that is brought into the market under the title "pure yttrium oxide," and in reality only consists partly of yttria, and contains as well oxides of nearly all the remaining rare earths.

We found, with the help of the absorption spectrum, 12—15 per cent erbium oxide; there were also slight indications of the oxides of neodymium and praseodymium; in working with larger quantities we later obtained fractions showing samarium lines. The hydrogen peroxide reaction showed traces of cerium; the cathode luminous spectrum contained the line in the orange characteristic of gadolinium; in short, all the rare earths that could be detected by the help of simple reactions were found to be present in our mixture in greater or less degree.

In order to determine the proportion of this oxide by the above described fractionation method, 40 grms. were pounded with about 90 grms. chromium trioxide, and introduced into 1 litre of water, whereupon, with violent action, solution into the dichromate resulted. The liquid was then treated with potassium chromate solution, till it began to get cloudy, and placed in a 2 litre retort, the whole being so worked so as to give six fractions, which were now examined separately.

Fraction I. *a*, obtained in two hours with 250 c.c. of 10 per cent potassium chromate solution, yielded only 1·1 grm. oxide, for the earth solution obviously yet contained polychromates. The chromates were quite dark brown, and the oxides much more darkly coloured than the original material. The absorption spectrum showed a distinct increase of erbium oxide; while a 10 per cent solution of the original did not show the red erbium bands, the latter were distinctly visible in the first fraction. The didymium spectrum was also strengthened.

Fraction II. *a*, obtained like the first, consisted of 34 grms. of light yellow, fine, crystalline needles, with straight extinction. The oxalates were coloured a beautiful deep pink, and gave 13·6 grms. oxide. In this case also was observed an increase of erbium oxide. The oxides contained about 20 per cent of it. They possessed a much deeper tint, extending more into the orange than the original oxides, which indicates increase in gadolinium.

By evaporation of the solution to the original volume of 1 litre,—

Fraction II. *b* was obtained, 10·5 grms. chromate, which yielded 6·04 grms. oxide. Crystallisation gave three different bodies:—

1. Fine yellow needles, as with Fraction II. *a*.
2. Orange-red, thick, many sided prisms, almost like potassium bichromate, strongly doubly refractive.
3. Shining crystalline crusts of uniform olive-green colour, apparently consisting of regular octahedra, weakly doubly refractive, which form about two-thirds of the fraction.

As the chromates of Fraction II. *a* yielded about 40 per cent oxides, and those of Fraction II. *b* about 58 per cent, we must conclude that the latter contained less water of crystallisation than the former, which is remarkable, for the temperature of formation of both salts was the same. Still more remarkable was the fact that the oxides of both fractions were scarcely distinguishable in colour and content of erbium.

Fraction III, was obtained with 300 c.c. of 5 per cent potassium chromate solution; 17·6 grms. precipitate, consisting of a quantity of the yellow needles and orange-coloured prisms. From this was obtained 7·04 grms. oxide, of colour almost the same as the original material. Erbium content about 10 per cent; didymium lines scarcely visible. On evaporation of the filtrate only small quantities of the chromate were obtained, which altogether gave 0·4 grm. oxide, and these were added to III.

Fraction IV. gave a similar quantity of two different kinds of crystals, treated just as III. The two varieties were separated mechanically from each other and carefully examined, with the result:—Orange-coloured prisms, 7·85 grms., yielding 2·55 grms. oxide, = 32·5 per cent; light yellow needles, 7·00 grms., yielding 2·8 grms. oxide, = 40 per cent. The yield of oxide was therefore less in this case than in the previous fractions, which indicates an increase in an earth of small equivalent, viz., yttria. In fact, both fractions exhibited a diminution of erbium; they contained only about 2·5 per cent of it. The oxalates appeared still only in small quantity. The solutions were no longer coloured. Also the content of gadolinium was diminished, to judge by the colour of the oxides. No material difference could be discovered with the spectroscope between the two oxides. On evaporation of the solution we obtained the dark orange coloured chromate of a still lighter earth; 7·5 grms. gave 2·065 grms. oxide, = 27·4 per cent. The oxide was almost pure white; the erbium spectrum in the solution only indicated.

Fraction V., carried out exactly as in the two former, yielded chromates of almost pure yttria directly, as well as by evaporation of the filtered liquid. These consisted of beautiful orange-coloured prisms; 4·2 grms. oxide were obtained directly, 1·5 grms. by evaporation. Colour of the oxide pure white; no trace of absorption spectrum, even in very concentrated solution.

Fraction VI. gave an oxide in almost exactly the same state, representing the remainder of the earths to be found in the solution, from which arose 1·5 grms. oxide, = 27·4 per cent. The precipitate was obtained from great excess of potassium chromate, 250 c.c. of a 10 per cent solution, in order to throw down all the earths. In this case also the precipitate was uniform, and consisted of deep red-coloured short prisms.

The remaining solution, from which nothing more was precipitated by potassium chromate, was treated with caustic soda; the precipitate contained magnesia, lime, silica, and traces of yttria.

(To be continued).

NOTE ON THE

ESTIMATION OF MANGANESE IN STEEL.

By HORACE JERVIS.

That process is referred to whereby the Mn is oxidised to permanganate in an acid solution with Pb_3O_4 or PbO_2. Perhaps no other process has been so often described under the heading "A New Process for Estimating, &c." It may be a dangerous matter to meddle with questions of priority, but it is certainly somewhat confusing to hear of Deshay's process, Peter's process, Chatard's process, Textor's process, and so on, when one is unaware that essentially one and the same process is meant.

Without doubt the process is a reliable one within very narrow limits, and there does not seem to be much to choose between the well-known modifications of it, except in special cases. I can speak with greatest confidence, however, of that modification in which the sample is dissolved in nitro-sulphuric acid, oxidised with red-lead, and the filtrate titrated with ferrous sulphate and permanganate.

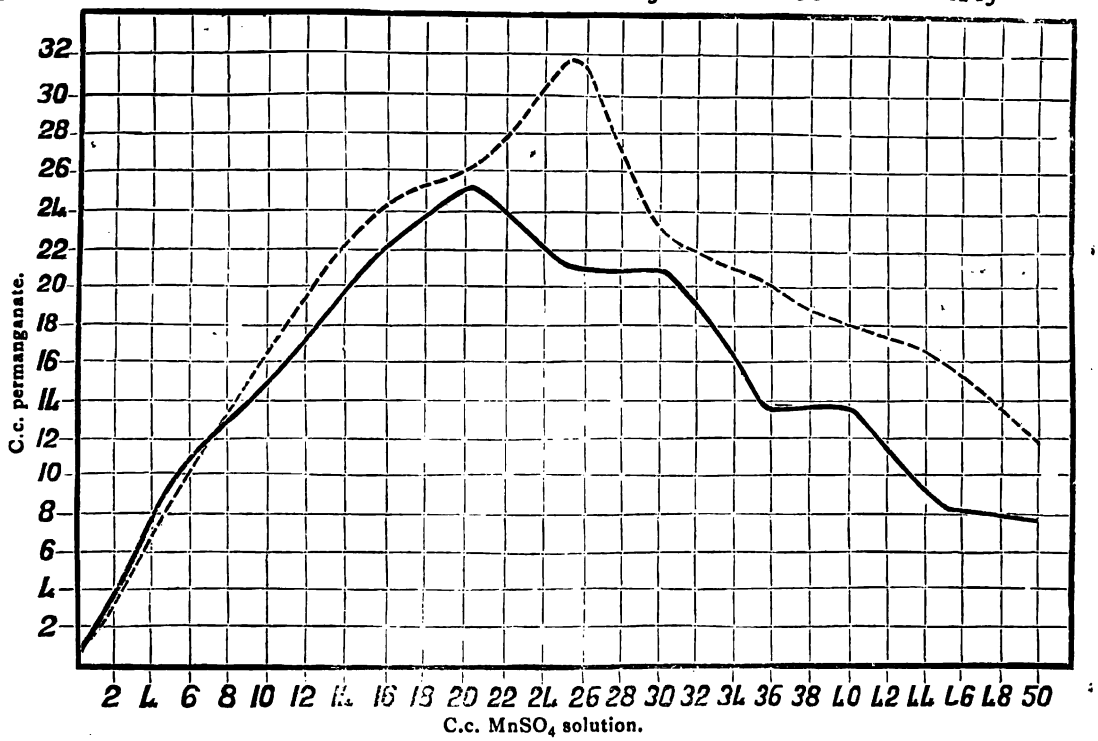
One per cent of manganese, using 1 grm. of the sample, is properly regarded as the extreme limit within which accurate results are obtainable, but samples up to 2 per cent can be assayed if ½ grm., or up to 4 per cent if 0·25 grm., is weighed off, and so on until a limit is imposed by the delicacy of the operation.

In the ordinary use of the process, the iron is distinctly favourable to the conversion of MnO into permanganate, —more permanganate is obtained with it than without it, —but either with or without iron the recovery of manganese falls appreciably short when somewhat less than 0·01 grm. manganese is present.

This deficiency augments very rapidly until we reach a point beyond which the more manganese one puts in the less one gets out. This conclusion limits Deshay's statement (Chemical News, xxxviii., 70) that the colour is stronger as the manganese increases, and is so unexpected that it seems worth while to illustrate it.

In one series of experiments 1 grm. of bar iron, with increasing amounts of manganese, was oxidised with red-lead in the usual way; another series was treated in the same way, but no iron was present. The amounts of manganous sulphate solution added and the permanganate found in the filtrate expressed in terms of a standard solution are shown in the table and plotted in the curve.

$MnSO_4$ solution added. C.c.	C.c. permanganate found. Iron present.	No iron.
2	3·45	3·35
5	9·84	8·5
10	14·8	16·45
15	21·0	23·55
20	24 7	25·85
25	21·15	30 94
30	21·0	22·9
35	14·6	20·94
40	13·75	18·0
45	8·3	16·2
50	8·0	12·15



Continuous line, with Iron. Broken line, without Iron.
2 c.c. $MnSO_4$ = 0·0045 grm. Mn.

This peculiarity of the process is more serious than at first sight it appears to be. It means that with perfectly strange material one has to decide whether the percentage recorded is that actually present, or a mere fraction of a much larger percentage. I am aware of an instance in which 0·40 per cent of Mn was returned when really 13 per cent was present, and a 20 per cent spiegel or an 80 per cent ferro-manganese, treated like an ordinary steel, shows not even the slightest trace of manganese. Permanganate is momentarily formed, however, just as the red-lead is added, and may be formed permanently by adding more and more red-lead.

Aluminium has no effect on this process; copper and nickel no further influence than is exercised by their colour in the filtrate. Molybdenum does not interfere. In fact, the process may be used for estimating small amounts of

manganese in commercial metallic molybdenum powders, if the precaution be taken to filter off associated tungsten and graphite. Tungsten steels may be safely assayed for manganese by this process, but grey pig-irons in our experience, even when the graphite has been filtered off, or otherwise destroyed, give somewhat lower results than the gravimetric estimation. Any chromium present is oxidised to chromic acid, and this interferes with the process more or less according to the substance used for titrating the permanganate. Ferrous sulphate cannot be used; it reduces bichromate almost as readily as it does permanganate when the two are present together.

When ammonium oxalate is used for titrating the permanganate the results are very satisfactory. The most convenient and at the same time accurate practice is as follows: Gauge the approximate percentage of manganese in the sample by the colour of the filtrate, run from a burette into the heated (70—80° C.) solution as much ammonium oxalate as will certainly destroy the colour without leaving a great excess, and go back as quickly as possible with standard permanganate. The colour changes from lemon to orange when only a trace of permanganate is in excess. The volume of solution titrated should be about 400 c.c. An excess of oxalate should be avoided as far as possible because it slowly reduces the chromic acid.

The chromic acid may now be titrated with ferrous sulphate for the estimation of the chromium, but the results are considerably low if several per cents chromium happen to be present. If the lead peroxide residue be treated with dilute hydrochloric acid the chlorine expelled by boiling, and that portion of chromic acid added to the results which was present in the residue, the results are sometimes as much as two-tenths per cent low.

A NEW COLOURED REACTION OF TYROSINE.

By G. DENIGÈS.

There are, up to the present time, four coloured reactions of tyrosine known; the first is due to Piria, the second to Hofmann, the third to Schérer, and the last to Bertrand. This latter one, of a bio-chemical order, has been specially studied and applied by Bourquelot and his pupils.

Piria's reaction is obtained by dissolving the tyrosine in hot concentrated sulphuric acid; in this manner tyrosine sulphuric acid is formed. This is allowed to cool, is diluted with water, neutralised with carbonate of barium, and filtered. The filtrate, to which ferric chloride is added, gives a beautiful violet colouration. The reaction does not take place in the presence of a mineral acid or on the addition of an excess of ferric chloride.

To obtain Hofmann's reaction we place a small quantity of tyrosine in a test-tube with 2 or 3 c.c. of water and 10 drops of Millon's reagent. This is brought to boiling, and kept at that temperature for a few moments; the solution takes a beautiful red colour, and becomes cloudy owing to the formation of a precipitate of the same colour.

We may also boil the tyrosine with mercuric nitrate, and then add nitrous nitric acid to the liquid.

Schérer's reaction is effected by the careful evaporation to dryness, on a sheet of platinum, of a few drops of nitric acid and tyrosine; in this manner a beautiful yellow residue is obtained, formed of nitrate of nitrotyrosine, which, on the addition of soda, takes a deep orange tint.

It is very evident that these two latter reactions are not practicable in the presence of albumenoid matter, which also causes them, as also do many other substances which behave in the same manner in the presence of Millon's reagent or hot nitric acid.

Piria's reaction, which is not very sensitive, and is difficult to perform, may also be obtained by a number of phenolic bodies, but it cannot be obtained in the presence of hydrate of carbon and of organic matters in general, which become brown on contact with warm concentrated sulphuric acid.

Of much greater interest is the Bertrand-Bourquelot reaction, which makes use of the property possessed by tyrosinase (a special oxydase contained by a large number of vegetables, especially mushrooms), of giving, with solutions of tyrosine, a red colouration which eventually turns black.

It suffices, in fact, to add 8 or 10 c.c. of the solution in which tyrosine is to be sought for, a few drops of an aqueous extract of *Russula delica*, previously ground with sand, to see the above-mentioned two successive tints rapidly appear.

The drawback to this beautiful reaction is the necessity of having tyrosinase handy, a substance which is not always easily to be obtained.

Finally, it must not be forgotten that Bourquelot has shown a large number of phenols; their ethers, and their corresponding amidised bodies, are also oxidised in the presence of tyrosinase, furnishing various colourations, of which some are often difficult to distinguish from that given by tyrosine under the influence of the same ferment.

The new reaction which we propose, and which we have discovered, is founded on the property possessed by tyrosine of giving with ethanal (ordinary aldehyde), in a strongly acid medium, a product of condensation of a beautiful rose-carmine colour, having a wide absorption band covering the green and nearly the whole of the yellow of the spectrum.

Ordinary aldehyde, which boils at about 21°, not being very convenient to handle, we used it diluted to one-third with alcohol at 90°, according to the formula—

Aldehyde	5 c.c.
Alcohol at 90°	10 ,,

For the detection of tyrosine we put in a test-tube 2 c.c. of pure sulphuric acid, and 3 to 5 drops of the alcoholic solution of aldehyde, added by means of a pipette. Care must be taken only to add the aldehydic solution drop by drop, every two or three seconds, shaking the liquid thoroughly after the addition of each drop, which must not be allowed to touch the sides of the tube, but must fall directly into the acid. If these several precautions are not taken we should run the risk of causing the formation of a brownish-yellow tint which would interfere with the subsequent observation of the reaction.

As soon as the mixture is made* we add a small quantity of tyrosine, and shake well; almost immediately the mass takes a more or less decided red colour, according to the quantities present and of an intensity proportional, within certain limits, to the quantity of tyrosine present in the sample.

Thus, by dissolving 1 c.grm. of tyrosine in 1 drop of soda lye and 5 c.c. of hot water, 1/20th of a c.c. of this solution containing 1/10th of a m.grm. of tyrosine, gave a beautiful red tint with the aldehydic reagent.

With a solution diluted five times, and a sample corresponding to 1/50th of a m.grm. of tyrosine, the tint was still very decided; it was still appreciable when using a solution of 1/10th the strength of the first, which corresponds to 1/100th of a m.grm. of the product in the sample taken, or 0·2 grm. per litre.

As it is easy to use 1/10th of a c.c. of the tyrosinic solution for each 2 of the sulpho-aldehydic reagent, it will be seen that it is possible to detect tyrosine directly in a solution not containing more than 10 c.grms. per litre.

By making a comparative experiment with a titrated tyrosinic solution, it is possible, by the aid of this reaction, to make a rapid colorimetric estimation of tyrosine, not only in aqueous solution, but also in the liquors of pancreatic digestion; albumenoid matters and peptones (free

* This mixture should not be made until the moment it is wanted, as it changes rapidly. In its preparation we can, if necessary, replace the aldehyde by paraldehyde or metaldehyde.

from tyrosine) do not influence the sulpho-aldehydic reaction.

Finally, we can utilise it for the study of the elimination of urinary tyrosine, by concentrating the urine when this substance is present in smaller quantity than 0·10 or 0·20 grm. per litre.

Although this reagent is influenced by certain phenolic products or their ethers, these compounds have no effect except when present in much greater quantities than in the case of tyrosine, and, further, they do not exist at all in most cases when tyrosine is to be sought for.

Formolised sulphuric acid, prepared by adding 1 c.c. of commercial formol to 50 c.c. of pure sulphuric acid, and which constitutes a very stable reagent, is, contrary to precedent, more sensitive with bodies of the phenolic function than with tyrosine. We hope to use it in the diagnosis of urinary phenols.

When, to 2 c.c. of this reagent, we add a little tyrosine, a colouration of dead leaves is developed, slowly in the cold, but instantaneously at 50—60°, finally taking a reddish tinge.

By adding to the liquid double its volume of glacial acetic acid and boiling, the tint changes to green.—*Bull. des Trav. de la Soc. de Pharm. de Bordeaux*, Feb., 1900.

ON THE CADMIFEROUS-ZINC ORES.

By E. JENSCH.

In most treatises on metallurgy it is stated that the zinc ores of Silesia contain from 2 to 5 per cent of cadmium, but the author has found that the ore at present taken from these mines only contains from 0·04 to 0·306 per cent, or an average of 0·102 per cent.

The blende from the upper Harz district, also reputed to contain a high proportion of cadmium, does not, however, contain more than 0·01 per cent at the most, and sometimes this element is entirely absent.

In all the Saxony ores, the proportion of cadmium present varies from 0·011 to 0·100 per cent; in those from Styria and Carinthia, from 0·003 to 0·140 per cent.

The highest proportion of cadmium—0·46 per cent—was found in a parcel of 208 tons of black blende from Finland; but the blende from Matlock and Wanlockhead, as well as the transparent yellow variety from Cunillas, near Santander, also contains more than 0·40 per cent.

In the Swedish and Norwegian blendes the proportion varies from 0·047 to 0·40 per cent. The poorest varieties are those from the north of Norway; they only contain 0·008 to 0·010 per cent of cadmium.

Cadmium is also met with in coal, where it probably exists in combination with pyrites. The highest proportion found was 0·008 per cent in a raw coal; the same coal, washed, contained no more than 0·001 per cent.

A large proportion of the cadmium contained in the zinc ores is lost during calcination. The mean, 0·110 per cent, in the Silesian blendes, falls to 0·042 per cent after roasting. The cadmium is partly condensed with the flue dust, which contains from 0·22 to 2·02 per cent.—*Berg.-u. Hüttenmännische Zeit.*, 1899, p. 14.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 2nd instant, Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. The following were elected Members:—Messrs. R. T. Glazebrook, E. J. Humphrey, H. S. Maxim, S. W. A. Noble, W. F. Snell, W. J. Tennant. It was announced that the Actonian Prize of 100 guineas had been awarded to Sir William and Lady Huggins for their work "An Atlas of Representative Stellar Spectra." The special thanks of the Members were returned to Mrs. West and Mrs. F. Colenso for their present of a portrait of their father, the late Sir Edward Frankland, K.C.B., D.C.L., F.R.S., Professor of Chemistry of the Royal Institution from 1863 to 1868.

ON THE MANUFACTURE OF CARBIDE OF CALCIUM.

By G. HANEKROP.

1. *The Electric Conductivity of Melted Carbide of Calcium.*

As this subject does not appear to have been examined, it seems of interest to record the results I obtained from a number of experiments. The measurements were made in the following manner:—

The block of carbide rested on the lower electrode of the furnace; this latter was connected with a copper wire. Further, at two places in the block, through the solid crust to the part remaining fluid, two rods of iron were inserted to which copper conductors were connected. It was thus possible to measure the fall of potential in the lower layers of the block, through a distance of 8 or 10 c.m., and also that between the iron rods to a variable distance. In Table I. we give a part of the results obtained. The figures relating to the intensity of the current and to the fall of potential were obtained by means of ten readings with variable intensities. For measuring the difference of potential we made use of a very sensitive Weston voltmeter reading to $\frac{1}{100}$th of a volt. The ammeter used, also by Weston, was graduated up to 1000 ampères; thus measurement could not be made beyond these limits.

It can be seen (column 9) that the resistance of melted carbide of calcium is from 430 to 630 times that of mercury; the conductivity varies from $\frac{1}{430}$th to $\frac{1}{630}$th.

These variations should be attributed to the differences of temperature in the interior of the block; we are unable, however, to measure these temperatures.

On comparing the values of columns 3 and 6, we notice that the block is of a higher resistance at its lower part; this is accounted for by the fact that when the block has reached a certain height the base no longer remains liquid, but pasty, thanks to the heat still received by conduction or radiation.

This fall of potential in the base of the block occurs to the same degree both in continuous and intermittent manufacture. It is well known that, by the first method, the carbide is run off at certain intervals of time, and that the production is continued in this manner for several days; while, by the second method, the carbide is left in the furnace by constantly raising the upper electrode, so as to obtain a block of carbide. This block is not removed from the furnace until it has sufficiently cooled and solidified.

Now, as the fall in the lower part is already greater than that in the rest of the block, the economy thus obtained—which was looked upon as the principal advantage the continuous process had over the intermittent one—does not appear to be very great, and should be largely counterbalanced by the loss of heat resulting from each running off of the carbide. Further, in the continuous process, it is always necessary to keep a block of a certain thickness, no matter how little, for the reaction to be regular. There is therefore at the base always a layer of more or less solidified carbide, the electric resistance of which is relatively high.

2. *The Use of Wood-charcoal in the Manufacture of the Carbide.*

The opinion has often been expressed that wood-charcoal is not suitable for the manufacture of carbide of calcium; and this opinion could even recently be seen in print in the *Zeitschrift f. Calcium Carbid Fabrikation*, ii., 416; and iii., 19. A practical test of sufficiently long duration is enough to refute this quite erroneous opinion. For a year and a half wood-charcoal, and especially that obtained from the distillation of wood at the Actiengesellschaft für Trebertrocknung, at Cassel, has been found to make an excellent raw material.

On an average, 780 kilogrms. of this wood-charcoal was

Table I.

	Measurements between the lower electrode and Rod 2.			Measurements between Rods 2 and 3.					
No	Ampères.	Volts.	Ohms.	Ampères.	Volts.	Ohms.	Distance in decimetres between points 2 and 3.	Section of block in decimetres.	Ohms per 1 decimetre in length and of 1 decimetre section
	1.	2.	3.	4.	5.	6.	7.	8.	9.
1.	890	2·14	0·00240	888	0·78	0·00087	1·40	10·17	0·00632
2.	942	1·82	0·00193	974	0·85	0·00088	1·60	11·34	0·00624
3.	876	2 62	0·00300	860	1·40	0·00262	2·00	7·07	0·0 ,572
4.	808	1·90	0·00235	922	1·98	0·00215	3·50	9·07	0·0·1557
5.	806	2 04	0·00250	762	1·19	0·00163	4·00	10·74	0·00429

used to obtain 1000 kilogrms. of carbide. The utilisation of the electric energy is very high; we can easily obtain 4 to 4·5 kilogrms. of carbide per 24 h.-p. hours; with careful working we have even obtained 4·5 to 4·7, giving about 300 litres of gas per kilogrm. The best results and the best utilisation of electric energy are obtained with wood-charcoal rather than with coke, and depend perhaps on the fact that, at the temperature of the arc, the former is more easily transformed into graphite than the latter. Wood charcoal also reacts more easily than coke.

Carbide prepared with wood-charcoal gives a very pure acetylene. The following figures will serve as terms of comparison. There were found in 1 cubic metre of acetylene—

Table II.

	Acetylene from carbide prepared from wood-charcoal.		Acetylene from carbide prepared from coke from various sources.		
	a.	*b.*	*a.*	*b.*	*c.*
	C.c.	C.c.	C.c.	C.c.	C c.
PH_2 ..	243	199	236	380	456
H_2S ..	73	Not estimated	505	457	Not estimated

If the carbide prepared from wood-charcoal is decomposed by a sufficient quantity of water, it can be used for lighting purposes without further purification. This carbide is also very suitable for portable lamps to which a system of purification cannot be adapted.

3. *Analysis.*

The determination of the impurities in acetylene gas by Lunge and Cedercreuz's method is done by means of hypochlorite of soda; but, instead of using a tube with ten bulbs to hold the hypochlorite solution, we used two glass tubes 15 m.m. in diameter and 700 m.m. long, simply curved, and penetrated at each end by a small tube. The funnel tube, with a tap, should be about 30 c.m. long, and the reservoir should have a capacity of about 150 c.c. This volume of water suffices for the decomposition of 50 to 55 grms. of carbide of calcium.

We may combine with the determination of the impurities of the gas that of the return of acetylene from the carbide. For this purpose we insert a chloride of calcium tube. We need then only proceed as with a Bunsen apparatus for the estimation of carbonic acid—an apparatus so well known that it is unnecessary to enter into details. From the loss of weight caused by the disengagement of acetylene we calculate the return in gas. One litre of acetylene weighs 1·161 grm. at 0° and 760 m.m. B.P.

Water is used for the decomposition of the carbide; it is useless to add any salt whatever to diminish the rapidity of the reaction. We can almost entirely prevent the formation of steam by placing the flask used for the analysis in a dish full of cold water. Five or six drops of water a minute are let run over it. As nothing but water is used for the decomposition of the carbide, the residue can be used for future analysis.

We must here mention that the globules of ferro-silicon which are sometimes met with in carbide are easily soluble in a mixture of sulphuric and hydrofluoric acids, while they are almost completely insoluble in hydrochloric, nitric, and sulphuric acids. Carbide of silicon, which is also found with carbide of calcium, is decomposed by fusion with a mixture of carbonate and peroxide of soda.—*Zeitschrift für Angewandte Chemie*, 1899, p. 592.

A REVISION OF THE ATOMIC WEIGHT OF IRON.*

(Preliminary Paper).

By THEODORE WILLIAM RICHARDS and GREGORY PAUL BAXTER.

It is rather surprising that the atomic weight of a metal so important as iron should have received so little recent attention as it has. Berzelius† was among the first to determine the quantity under discussion; and in order to accomplish the determination he converted Swedish pianowire into ferric oxide. For many years his very low results, 54·3, was almost unchallenged, when in 1843 Wackenroder called attention to the fact that in some early analyses of ferric oxide Stromeyer and he had found the substance to contain only 30·1 per cent of oxygen, corresponding to an atomic weight of nearly 56. This led immediately to several investigations by Svanberg and Norlin, Berzelius, and Erdmann and Marchand, whose individual results upon the same ratio varied from 55·81 to 56·23, showing without a doubt that the low value was incorrect, and settling the question with all the accuracy necessary in that day. Rivot in 1850 published two poor analyses which shed no further light upon the subject; and the more recent work of Dumas, who analysed the chlorides of iron in 1859, was no more accurate than his similar work on other metals. Since that time no work on the atomic weight worthy of notice has appeared, so that our present knowledge depends upon data obtained over fifty years ago. In the intervening time Stas has taught the world accuracy in chemical analysis, and countless complications and sources of inaccuracy have been discovered which then were unsuspected. The variations of nearly 0·8 per cent in the best determinations (all depending upon the composition of ferric oxide), point with emphasis toward the necessity of a modern determination. Such questions as the possible presence of gases in the oxide, the possibility of incomplete reduction, and the danger of contamination from the containing vessels, should obviously receive attention.

The balance and weights used in the present study were the same as those used in other recent determinations of atomic weights (*Proc. Amer. Acad.*, 1891, xxvi., 242). It is needless to state that the weights were carefully justified, and that all the usual precautions necessary to procure accuracy in such work were observed. All the weighings were reduced to the vacuum standard, and the

* Contributions from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxv., No. 13.

† The references to all the sources of information are to be found in the critical discussion at the close of this paper.

values of the atomic weights refer to the universally accepted standard O = 16·000.

One of the most important steps in such an investigation is clearly the choice of the substance to be analysed, and the choice usually requires a careful preliminary qualitative and quantitative study of many compounds of the element under consideration. This work occupied a number of weeks, and in the course of it we found that the preparation of either ferrous or ferric halides in a pure anhydrous state offered so many difficulties that as yet no satisfactory method for the purpose has been devised. Ferric oxide, on the other hand, can be obtained in a variety of ways, hence the choice of the early experimenters seems to have been a wise one. The first step in a systematic treatment of the subject is obviously a repetition of the discordant work of half a century ago, with especial reference to the possible errors noted above.

Owing to the fact that most oxides formed from nitrates are known to include considerable quantities of oxygen and nitrogen (Richards, *Proc. Amer. Acad.*, 1891, xxvi., 281; Richards and Rogers, *Proc. Amer. Acad.*, xxviii., 200), ignition of ferric nitrate as a means of obtaining ferric oxide was at first rejected, and ferric hydroxide was used instead. Very pure iron ribbon was dissolved in sulphuric acid, and the metal was then deposited electrolytically from an ammonic oxalate solution. After the film had been dissolved in the purest nitric acid, the carbon was removed by filtration, and the iron was precipitated as ferric hydrate by the addition of an excess of ammonia which had been distilled into pure water contained in a platinum dish. The precipitate was washed thoroughly, collected upon a pure washed filter paper, separated from the filter paper while moist, and finally dried in a platinum dish upon the steam bath. The lumps of dried hydrate were crushed in an agate mortar and finally ignited for several hours in a current of pure dry air. During the latter operation the weighed platinum boat with the oxide was contained in a large porcelain tube heated by a Fletcher furnace to about 900°. Constant weight within a few hundredths of a milligrm. was obtained without difficulty. The weighed oxide was then reduced in a current of electrolytic hydrogen which had been dried by means of fused potassic hydroxide. Ferric oxide is only very slowly reduced at the highest temperature which can be used with glass tubes, so that this reduction also was of necessity carried on in porcelain tubes. Even at about 900°,* long continued heating was needed to complete the reduction, the total period of ignition amounting sometimes to twenty hours. The progress of the reaction was slightly accelerated by alternate oxidation and reduction.

Two analyses, carried out in the fashion described, are tabulated below. Corrections to a vacuum standard have been applied by adding 0·00009 grm. to every apparent grm. of ferric oxide, and 0·00001 grm. to every apparent grm. of metallic iron, the specific gravities of these two substances being assumed to be 5·2 and 7·9 respectively (Landolt und Börnstein "Tabellen," 1894, cxviii., 133).

Series I.

Number of analysis.	Weight of ferric oxide in vacuum.	Weight of iron in vaccum.	Atomic weight of iron.
1	3·17485	2·22096	55·885
2	3·61235	2·52750	55·916
	Average..		55·900

The fact that both of these results are considerably lower than the accepted value of the atomic weight of iron (56·0) seemed to confirm a suspicion which had already arisen that ferric hydrate might not be completely converted into the oxide by ignition at a high temperature. That the oxide upon ignition easily reached constant weight is, however, evidence in the other direction. Since no direct method of proving the presence or absence of retained water was at hand, it seemed best to make use of another method of preparing ferric oxide. Although, as has been already stated, oxides formed from nitrates occlude nitrogen and oxygen, yet these gases are evolved upon solution of the oxides and can be determined. Ignition of ferric nitrate was therefore chosen as the next method of obtaining ferric oxide.

The material used in the following work was purified with great care. In the first place a solution of a so-called "chemically pure" chloride of iron was treated with an excess of sulphuretted hydrogen and the resulting sulphides and sulphur were removed by filtration. From the filtrate, after oxidation with nitric acid, the iron was precipitated with an excess of ammonia hydrate and the precipitate was thoroughly washed by decantation. In this process traces of a number of metals which might have been present must have been removed. The precipitate was dissolved in sulphuric acid, and by cautious addition of ammonic hydrate basic ferric sulphate was thrown down; a precipitate which is much more easily washed than ferric hydrate, but which was re-dissolved in sulphuric acid with considerable difficulty. In order to reduce to the ferrous state the ferric sulphate thus formed, the solution was next subjected to the action of a galvanic current of several ampères. The solution was contained in a large platinum dish which served as the negative electrode, the positive electrode being a flat spiral of platinum wire. Since the solution was very concentrated and contained a considerable excess of sulphuric acid, the greater part of the ferrous sulphate crystallised out when the solution was allowed to cool. By alternately electrolysing and cooling, almost all of the iron was eventually obtained as ferrous sulphate.

The next process in the purification was to deposit the iron electrolytically from a solution of its oxalate. By means of this separation, the aluminum and manganese could be eliminated, since the first, together with alkaline and other impurities, is not deposited by electrolysis, while the second is deposited upon the anode. The ammonic oxalate needed was prepared with great care. Oxalic acid which had been repeatedly crystallised with hydrochloric acid, and then until free from chlorine, was saturated with pure re-distilled ammonia, and the resulting ammonic oxalate was subjected to several crystallisations. To a hot concentrated solution of this ammonic oxalate, the ferrous sulphate was added as long as no permanent precipitate resulted, and the solution was then electrolysed until the greater part of the iron had been deposited. A slight deposit of hydrated manganese peroxide appeared upon the positive pole, which, however, presented enough surface to prevent the deposit from becoming detached. The film of iron was carefully washed and dissolved in pure dilute nitric acid. When all of the ferrous sulphate had been thus converted into ferric nitrate, the solution of the nitrate was filtered and evaporated, and the salt was three times re-crystallised from a large amount of nitric acid. This acid had been distilled in a platinum still and condenser and collected in a platinum receiver. The mother liquor of the first crystallisation was analysed for impurities as follows: After precipitation of the iron as ferric hydrate by the addition of an excess of pure ammonia and filtration, the filtrate was exaporated to dryness and the residue was ignited. Since only a negligible amount of alkaline solid remained, it can fairly be assumed that the final preparation of ferric nitrate must have been free from such impurity. During the latter part of this purification platinum vessels only were employed, and the water and reagents were the purest obtainable.

The next step, that of converting the nitrate into oxide, was carried out in part by gentle heating in an electric oven at about 150° (Richards, *Amer. Chem. Journ.*, 1899, xxii., 45). The resulting cake of basic nitrate, after removal from the plat num dish in which it was contained during the above operation, was crushed in an agate mortar, and the reaction was completed by ignition in an

* At the temperature to which the porcelain tubes were heated pure silver did not melt, while sodic chloride was easily fused.

open porcelain tube at a temperature of about 900° for several hours. The oxide, which was now contained in a large platinum boat, was carefully weighed and again heated. The loss of weight during this second ignition amounted to one-tenth of a milligrm., while a third prolonged heating caused a further loss of three-tenths of a milligrm. This difference must have been caused by the escape of included gases, and was so small in comparison with the weight of the oxide (17 grms.), that further heating was deemed unnecessary.

The determination of the gases contained in this oxide proved to be a matter of considerable difficulty. At first the method used was that of dissolving the oxide in a Sprengel vacuum in fused acid potassic sulphate. Owing to the fact that the sulphuric acid, which was evolved from the fused sulphate, attacked both the rubber connections used for joining the apparatus, and also the stick potash used as a drying agent, it was uncertain whether the small quantity of gas which was collected in the air-pump was originally included in the oxide. A more successful method was eventually found by dissolving the oxide in hot hydrochloric acid containing stannous chloride. The operation was carried on in an apparatus for determining the gases evolved during the solution of a substance, which has already been described in papers from this laboratory (*Proc. Amer. Acad.*, xxviii., 200). In two experiments half a grm. of ferric oxide yielded a mere trace of gas. Cupric oxide treated in the same way evolved considerable gas, less, however, than that evolved when the same cupric oxide was dissolved in sulphuric acid. It was shown, however, that the gas which resulted upon solution in hydrochloric acid and stannous chloride consisted wholly of nitrogen, and that the volume of this nitrogen corresponded to the nitrogen in the gases set free by solution of the same oxide in sulphuric acid. Although it was evident that the oxygen was absorbed by the stannous chloride, yet, since in the case of all oxides heretofore examined the included gases consisted chiefly of nitrogen, it is fair to conclude that this was so in the case of ferric oxide. Only a trace even of nitrogen was evolved from the ferric oxide, hence this preparation must have been practically free from included gases.

In order to avoid any error due to hygroscopicity, the oxide used for each analysis was bottled in dry air in the bottling apparatus, which has already been described elsewhere (*Proc. Amer. Acad.*, 1896, xxxii., 59). The platinum boat containing the oxide was heated in the hard glass tube, while the air was exhausted from the apparatus by means of a Sprengel air-pump. Dry air was then admitted, and the boat was pushed into the weighing bottle, which was then stoppered and weighed with the usual precautions. Since in two instances repetition of the bottling failed to change the weight of the oxide, subsequently this precaution was omitted.

The oxide was then reduced in the manner already described, constant weight of the metal being attained much more easily than in the case of oxide prepared from ferric hydrate. Five closely agreeing results were obtained from analyses of material prepared as above.

Series II.

Number of analysis.	Weight of ferric oxide in vacuum.	Weight of iron in vacuum.	Atomic weight of iron.
3	3·97557	2·78115	55·883
4	4·89655	3·42558	55·891
5	4·35955	3·04990	55·891
6	7·14115	4·99533	55·870
7	6·42021	4·49130	55·882
	Average..		55·883

In order to determine if the reduction had been complete in the last two analyses of this series, the resulting metal was dissolved in cold dilute sulphuric acid. In each case a few tenths of a milligrm. of black insoluble matter remained, which contained platinum. This impurity probably came from the alloying of the boat with the iron during the reduction, and hence introduced no error. A trace of ferric oxide also was found. Even if the platinum had been originally present, the combined weight of the two impurities could not have raised the final result more than 0·01 per cent; hence no correction was applied.

One of us (Baxter, *Amer. Chem. Journ.*, 1899, xxii., 363), has recently shown that iron when ignited at a high temperature, has practically no tendency to occlude hydrogen, so that in the present case this possible source of error is excluded. On comparing these results with the two determinations of the first series, one is forced to conclude that the oxide made from the nitrate has essentially the same composition as the oxide made from the hydroxide. The higher value indicated by Experiment 2 was probably caused by incomplete reduction, and the first experiment agrees with the average of the second series.

It is interesting to review the older determinations (a complete list of which is given at the end of this paper) in the light of the experience gained in these analyses. Evidently the occlusion (or better, inclusion) of gases by the oxide is a less serious cause of error in the case of iron than in most other cases, if the temperature of ignition is reasonably high. This is the chief probable error which would tend to make the results too low. The three most probable errors having the opposite effect are:—First, the possible presence of magnetic oxide in the ferric oxide; second, the possibility of incomplete reduction during the analysis of this substance; and third, the possible presence of alkaline, siliceous, or other non-reducible material. Wackenroder seems to have been the only experimenter who thought seriously of the first two of these errors; and none of the early experimenters paid any attention to the last, for until recently glass was supposed by most chemists to be wholly insoluble. Wackenroder's work was perhaps the most intelligently carried out of all the older determinations, although his individual analyses did not agree among themselves quite so well as some of the others. His greatest omission was the recognised lack of purity in his hydrogen, although he could not observe an error due to this cause. It is interesting to note that the average of his six results gives the value 55·82 for the atomic weight in question, while a corrected average obtained by rejecting the two most discordant values gives the value 55·92. The result of the present work indicates a value midway between the two averages. Wackenroder's valuable work does not enter into Clarke's average for the atomic weight of iron, for Clarke was unable to find the original paper.

In order to prepare pure iron for his later work, Berzelius fused his metal with ferrous oxide, but gave no proof of the effectiveness of his treatment. It is not impossible that traces of the oxide may have been held by the iron, as copper dissolves cuprous oxide. Erdmann and Marchand made the ferric oxide, which they reduced quantitatively, by the ignition of ferrous oxalate—a method which invites the presence of magnetic oxide. Maumené's method was the process of filtration used in common analysis, in which the possible errors in both directions are plentiful.

These detailed criticisms, taken in connection with the general lack of accuracy which is to be observed in the quantitative work of half a century ago, seem to show that there is nothing improbable in the present result, 55·88. Of course the analysis of a single compound is never conclusive, so that this result is announced only as a preliminary one, which we shall hope to support or disprove in the near future.

List of Determinations.

		Fe=
1814	Wollaston, from Thenard's Results (*Phil. Trans.*, 1814, civ., 21)..	55·2
1826	Stromeyer, (*Pogg. Ann.*, 1826, vi., 471). Reduction Fe_2O_3 : Fe	55·5

		Fe=
1826	Berzelius (*Pogg. Ann.*, 1826, viii., 185). Oxidation $Fe_2 : Fe_2O_3$	54·3
1830±Wackenroder (*Archive Pharm*, 1843, xxxv., 279; 1843, xxxvi., 22). Reduction $Fe_2O_3 : Fe_2$		55·9
1844	Svanberg and Norlin (*Liebig's Ann. Chem. Pharm.*, 1844, l., 432; also *Berzelius Jahresbericht*, 1845, xxiv., 121; and 1846, xxv., 42):—	
	Oxidation $Fe_2 : Fe_2O_3$	55·87
	Reduction $Fe_2O_3 : Fe_2$	56·09
1844	Berzelius (the same references). Oxidation $Fe_2 : Fe_2O_3$	56·02
1844	Erdmann and Marchand (*J. Pr. Chem.*, xxxiii., 1; *Lieb. Ann.*, 1845, lii., 212). Reduction of oxide from oxalate $Fe_2O_3 : Fe_2$	56·02
1846	Maumené (*Ann. Chim. Phys.*, 1850, [3], xxx., 380). Oxidation $Fe_2 : Fe_2O_3$	56·00
1850	Rivot (*Ann. Chim. Phys.*, 1850, [3], xxx., 192). Reduction $Fe_2O_2 : Fe$	54·2
1859	Dumas (*Ann. Chim. Phys.*, 1859 [3], lv., 157; *Lieb. Ann.* 1860, cxiii., 26). Analysis of chlorides	56·2

The results of Magnus, Döbereiner, Capitaine (1841), and Torrey (1888), alluded to by Clarke, are not worthy of serious consideration. See Smithson, *Misc. Coll.*, "Constants of Nature," 1897, v., 287; also iv., 65 (Becker, 1880).

From some of these values Clarke (1897) computed the value 56·021, while Messrs. Landolt, Ostwald, and Seubert (1898) chose the number 56·0.

DETERMINATION OF CALCIUM AND MAGNESIUM IN ASHES.*

By J. K. HAYWOOD.

THOSE who have had any considerable number of ash samples to analyse according to the method adopted by the Association of Official Agricultural Chemists, cannot help having been struck with the extreme difficulty of determining calcium and magnesium. The whole trouble consists in washing the voluminous precipitate of basic acetate of iron and phosphate of iron, which is not only so bulky as to be troublesome, but also commences to run through the filter soon after washing is begun.

The following work was undertaken with the idea of showing that this washing can be entirely done away with, without seriously affecting the accuracy of the results. To do this, the precipitation of the phosphoric acid and iron from the solution of calcium and magnesium was made in a 500 c.c. flask, the solution then cooled to room temperature, made up to the 500 c.c. mark, and *well shaken*. 250 c.c. of this solution were then filtered off through a dry filter, keeping the funnel covered to prevent evaporation, evaporated to a small volume, ammonia added to get rid of any small traces of iron that might have dissolved in the liquor (nothing like the large amount that comes through the filter when the precipitate is washed), and the solution again filtered. The manganese is removed with bromine and ammonia, and calcium and magnesium determined in the filtrate. It will be noticed that this method does not correct for the volume occupied by the precipitate of basic acetate of iron and phosphate of iron, and it was to show that this correction is immaterial that the following work was undertaken.

For purposes of analysis three powders were prepared: one containing approximately 10 per cent of lime, 3% per cent of magnesia, and 86·5 per cent of sand; two containing 20 per cent lime, 5 per cent of magnesia, and 75 per cent of sand; and three containing 30 per cent of lime, 9 per cent of magnesia, and 61 per cent of sand. Ten grms. of each powder were dissolved in hydrochloric acid and filtered to a volume of 500 c.c., aliquot portions being taken for analysis.

Analyses of the above powders showed the following per cents of calcium and magnesium oxides:—

	I.	II.	III.
Lime	9·81	19·74	29·78
	9·85	19·66	29·86
Average.. ..	9·83	19·70	29·82
Magnesia	3·69	5·28	8·90
	3·71	5·26	8·90
Average.. ..	3·70	5·27	8·90

Aliquot portions were again taken from the solutions prepared as above, and phosphoric acid was added to each portion, amounting in the case of Solution I. to 5 per cent of the total amount of powder used, in the case of Solution II. to 10 per cent of the total amount of powder used, and in the case of Solution III. to 12 per cent of the total amount of powder used. Enough ferric chloride was added to precipitate all phosphoric acid, and these portions analysed according to my method mentioned above, with the following results:—

	I.	II.	III.
Lime	9·80	19·72	29·52
	9·76	19·80	29·54
Average.. ..	9·78	19·76	29·53
Magnesia	3·75	5·21	8·95
	3·82	Lost	8·93
Average.. ..	3·79	5·21	8·94

Showing practically the same results as those obtained above when no volume of a precipitate was neglected.

In the case of Solution III., a rather extreme case was next tried by adding enough phosphoric acid to correspond to 20 per cent of the original powder. The results were:—

	III.		
Lime	29·74	Magnesia	9·19
	29·90		9·21
Average ..	29·82		9·20

In my work last year on the official ash sample, which contained 9·83 per cent of phosphoric acid, the calcium and magnesium were determined according to the above method with the following results, in triplicate:—

Lime	11·94	Magnesia	5·83
	11·94		5·75
	11·97		5·77
Average ..	11·95		5·78

As against an average of lime 11·62, magnesia 5·74, by the other chemist engaged in the work.

The results obtained, arranged in tabular form, were:—

	Lime.	Magnesia.
No. 1.		
Before adding phosphorus pentoxide ..	9·83	3·70
After adding 5 per cent phosphorus pentoxide	9·78	3·79
No. 2.		
Before adding phosphorus pentoxide ..	19·70	5·27
After adding 10 per cent phosphorus pentoxide	19·76	5·21

* Contribution from the Division of Chemistry, U.S. Department of Agriculture. From the *Journal of the American Chemical Society*, vol. xxi., No. 6.

	Lime.	Magnesia.
No. 3.		
Before adding phosphorus pentoxide ..	29·82	8·90
After adding 12 per cent phosphorus pentoxide	29·53	8·94
After adding 20 per cent phosphorus pentoxide	29·82	9·20
Official Ash.		
Washing precipitate	11·62	5·74
Not washing precipitate..	11·95	5·78

In the case of ashes which contain a larger per cent of phosphoric acid than that included in the limits of this paper, a small correction may be necessary for the volume occupied by the precipitate. On account of lack of time I will reserve the investigation of this subject for a future paper.

THE

DETERMINATION OF ANTIMONY IN ORES.*

By THOMAS BROWN, Jun.

THE object of this paper is not to present new methods, but, on the other hand, the application of some very old ones which have been agreed upon by the buyers and sellers for determining antimony in ores, and which after trial have been proved to give more closely agreeing and satisfactory results than those formerly obtained by the many more widely used volumetric and gravimetric methods. In addition such observations and innovations in using these methods will here be mentioned as have been made by the writer in an experience of a year or so in analysing the Mexican ores for the large exporters. Only a limited number of commercial chemists, both on this continent and in Europe, are now engaged in doing this kind of work, because of the past wide variation in results frequently reported on the same sample, caused doubtless by the use of methods not so applicable to this class of ores, which is a matter of much importance considering the large shipments made from Mexico. The following list is of interest in showing the percentages of antimony reported on the same samples of Mexican ores by three prominent English commercial analysts before definite methods had been settled upon, and also the necessity for such a procedure. A, B, and C represent the results of these three chemists, B and C being of London, and D the percentage finally agreed upon in settling.

Sample No.	A.	B.	C.	D.
I.	59 96	52·25	56·1	55·6
II.	55·8	53·62	52·0	52·8
III.	57·27	54·02	54·2	54·1
IV.	58·13	54 5	54·8	54·6
V.	60 78	59 23	63 1	60·0
VI.	57·45	53·7	53·6	53 6
VII	—	53·5	54·4	53·9
VIII.	59 8	53·2	54 9	53·5
IX.	53 2	52·0	57·8	52·6
X.	—	50·4	51·5	51·9
XI.	—	52·4	53 7	53·0
XII.	—	51·7	53·2	52·4
XIII.	—	46·03	47 7	46·8

These ores are known to the trade as "clean ores," it being of rare occurrence to find impurities in the grades shipped other than silica, iron, and lime, which obviously would not interfere with the schemes of analysis used. The metals of the fifth and sixth groups sometimes found accompanying antimony in ores, such as lead, mercury, copper, tin, and arsenic, when found in quantities larger than traces, should be separated from the antimony when it is to be determined by the method used for sulphides, and also tin and arsenic when the antimony is to be determined by the oxide method. Shipments of ore from an unknown locality should consequently be put through a series of preliminary tests, and it is always advisable to test every sample for the most common impurities such as lead and arsenic; qualitatively for the purpose of modifying the sulphide method, and quantitatively if found in appreciable quantities, because of having to be taken into consideration in estimating the market value of the ore.

Lead is determined both qualitatively and quantitatively as the orange-yellow chromate ($PbCrO_4$). For the qualitative test the finely ground ore is boiled with concentrated nitric acid for several minutes, and if a sulphide, until the sulphide of antimony is entirely converted into the insoluble white oxide, then cooled and diluted. Ammonia is added in excess, then acetic acid in excess, and the whole heated for several minutes in order to bring the lead salts thoroughly into solution. The insoluble matter is now filtered off, and the filtrate treated with an excess of potassium bichromate, which precipitates the lead as chromate from this ammonium acetate and acetic acid solution. The lead can also advantageously be determined quantitatively in this form from the sulphide precipitate remaining on the filter after separating the antimony from the metals of the fifth group, as will be explained later in the scheme of analysis.

For arsenic a combination of the Canby and Mohr methods is used. One grm. of the finely powdered ore is mixed in a porcelain crucible with 8 to 10 parts of a mixture of equal parts of sodium carbonate and nitre, gradually brought to fusion over a burner or in a muffle, and kept so for five minutes or more. The fused mass is allowed to cool, disintegrated in hot water, and the insoluble matter filtered off. The filtrate is now acidified with nitric acid, and the carbonic and nitrous acid gases boiled off. An excess of an emulsion of zinc oxide, so apparent in the bottom of the beaker, is added, and should an unusually heavy precipitate of gelatinous silica and alumina be produced, a second filtration is necessary, also an extra addition of zinc oxide to the filtrate before the precipitation of the arsenic. This is precipitated as the reddish brown silver arsenate (Ag_3AsO_4) by the addition of silver nitrate in slight excess accompanied by vigorous stirring.

The precipitate mixed with zinc oxide is filtered, washed with cold water until the washings show no silver reaction, and dissolved off the filter with hot dilute nitric acid, catching the solution and washings in a beaker free from chlorine. When cold, this is titrated for silver with an empirical solution of potassium or ammonium thiocyanate, using 1 c.c. of a saturated solution of ferric sulphate as indicator, and titrating to the same pale amber-yellow colour as was obtained in standardising the solution against pure silver. For the standard silver solution 1 grm. of the pure metal is dissolved in nitric acid, boiled to expel red fumes, and diluted to 100 c.c. For the standard thiocyanate solution 9 and 3/10ths grms. of potassium thiocyanate or 7 and 6/10ths grms. of ammonium thiocyanate are dissolved and diluted to 1 litre; then 1 c.c. = 0·010 grm. silver, and 108 silver = 25 arsenic. This same treatment somewhat modified is used in testing the ore qualitatively for arsenic. After fusion, the addition of acid, boiling, and the addition of zinc oxide in excess, the whole is filtered, and the neutral filtrate tested for arsenic as before.

The antimony ores of Mexico most common to the trade are those of stibnite (Sb_2S_3); cervantite (Sb_2O_4) produced by the oxidation of the former; oxysulphides, mixtures of these two with a preponderance of the former; and senarmontite (Sb_2O_3), which is, however, of much rarer occurrence. The composition of the oxide may be inferred from its behaviour when boiled with concentrated hydrochloric acid, with the subsequent addition near the end of the treatment of a few drops of nitric acid. Cervantite

* From the *Journal of the American Chemical Society*, vol. xxi., No. 9.

treated thus is but sparingly soluble, while senarmontite is almost if not completely so. Stibnite is also completely soluble; if, however, the ore be an oxysulphide, as is frequently the case, insoluble oxide of antimony is found in the siliceous residue which is afterwards determined by fusion.

The following analysis of an ore from the State of Oaxaca illustrates this statement in regard to the solubility of these oxide ores: Moisture, 0·3; silica, 8·5; iron oxide and alumina, 1·6; iron pyrites (FeS_2), 0·9; lime and magnesia, traces; cervantite (Sb_2O_4), 3·8; senarmontite (Sb_2O_3), 84·7; total, 99·8. The results of a complete analysis being calculated to their probable composition, left a difference of 88·7 per cent, to be filled by the 70·8 per cent soluble and the 3 per cent insoluble antimony found, and which were calculated to the composition of senarmonite and cervantite as they were shown to be by their solubility.

Upon the behaviour of these ores when treated with concentrated hydrochloric acid plus a few drops of acid and boiling, is based their division into the following three classes for applying the schemes of analysis:—1. The soluble ores—stibnite (and senarmontite); 2. The practically insoluble ores—cervantite; 3. The partially soluble ores—the oxysulphides, stibnite mixed with cervantite.

(To be continued).

NOTICES OF BOOKS.

Elementary Practical Chemistry. By A. J. Cooper, B.A., B.Sc., F.I.C. With 44 Illustrations. London and New York: Whittaker and Co. 1899. Pp. 86.

This text-book is intended principally for the use of Students in Science Classes and Schools of Science, and for those who are engaged during the day time and are attempting to pursue their studies during the evening.

The theoretical bearing of the experiments described is only dealt with in outline, the book being merely a laboratory companion, in which the practical directions are made as explicit as possible.

We must again refer to a fault which is common to many elementary books on chemistry, in which some of the illustrations are trivial, and could well be dispensed with; this book is no exception, and would not lose by many omissions.

The Value of Electrical Treatment. By Julius Althaus, M.D., M.R.C.P., Lond., &c. Third Edition. London, New York, and Bombay: Longmans, Green, and Co. 1899. Pp. 165.

Though still viewed with a certain amount of suspicion and mistrust, there is no doubt that the use of electricity for medical purposes is gradually becoming more and more understood, and is now a recognised branch of study and teaching in most hospitals, while in the United States and Germany we read "almost every practitioner makes daily use of both the constant and intermittent current in his treatment of disease." The author, having been in intimate touch with this subject during a long professional career, has once more put forward his views on the uses and limits of usefulness of electricity in medicine.

There are two principal forms of electric energy at present utilised in medicine:—

I. Frictional, or static, electricity.
II. Current electricity.

This latter kind comprehends no less than six different varieties, such as Galvanism, the constant current from the main; Faradism; D'Arsonval's sinusoïdal current; D'Arsonval's current of high frequency and potential, by which the patient may be traversed by a current of 3000 milliampères at the rate of 500,000 to 1,000,000 oscillations per second; and Morton's static induced current.

After fully describing the apparatus necessary, the author considers the physiological effect of the different kinds of current, both locally and on the brain and nervous system in general.

He then describes the separate electrical treatment of a large number of definite diseases, giving full instructions as to the kind and amount of treatment required.

Though the author must be regarded as something of an optimist, we recommend a perusal of his work not only to the younger medical men who will be most likely to take advantage of its teachings, but also to those to whom it comes more or less as a revelation of ideas smacking somewhat of quackery.

Sugars and their Principal Derivatives. ("Les Sucres et leurs Principaux Dérivés"). By Prof. L. Maquenne. Paris: G. Carré et C. Naud. 1900. Pp. 1032.

The history of sugars forms at the present time a whole chapter in chemistry, which is no less important from the theoretical than from the descriptive or practical point of view; it is, in fact, on the researches of M. E. Fischer that the speculations of isometry in space have found their most solid basis, and the doctrine of asymmetric carbon its strongest confirmation.

Unfortunately the superabundance of facts, with all their accompanying developments, has prevented them being properly studied in any general treatise on chemistry. On the other hand, the work done on sugars is so widely spread over so many pamphlets and journals, that the bibliography of the subject has become very complex. The author has, for these reasons, endeavoured to unite in a systematic manner all the knowledge we now possess on this subject, which has so long occupied his attention.

The result of his labours, which we have now before us, gives eloquent testimony to the careful and systematic manner in which he has accomplished his object.

The volume is well printed and bound, besides being provided with a large index, and will make a handsome and useful book of reference.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 12, March 19, 1900.

Hydrates of Barium Peroxide.—M. de Forcrand.—Several hydrates of barium peroxide are known, among which the most important are $BaO_2\,H_2O$, $BaO_2.10H_2O$, BaO_2H_2O, $BaO_2H_2O_2$. The author continues the work announced in a previous paper on the examination of these compounds, preparing them by the addition of dilute hydrogen peroxide to the dissolved baryta in varying proportions. In each case he measures the heat evolved. When a large excess of hydrogen peroxide is used, compounds are formed which are further oxidised than the trioxide, but they are more and more unstable; they are, besides, very highly hydrated, the number of molecules of water having scarcely diminished, even in the last case.

Separation of the Rare Earths.—R. Chavastelon.—M. Urbain has recently shown, in a paper on the separation of the rare earths, how easily thorium and cerium can be separated from lanthanum and didymium. He shows that, in a mixture of these four substances, first thoria is eliminated, and then cerium. The author has been able to obtain satisfactory results by an inverse method—keeping thoria alone, or thoria and ceria, in solution,

whilst producing with the other rare metals compounds insoluble under the conditions of the experiment. Into an excess of hot or cold saturated solution of neutral sodium sulphite the neutral solution of the mixed earths is poured; cerium, lanthanum, and didymium are precipitated in the form of sulphites insoluble in excess of alkaline sulphite, practically the whole of the thoria remaining in solution. The insoluble sulphites are transformed into chlorides by the addition of HCl. The hydrates are precipitated by ammonia, well washed, and then brought in contact with excess of alkaline bicarbonate, the whole being stirred by a current of carbon dioxide; cerium peroxide alone dissolves. The author also describes another method of separation which is not quite so successful.

Chemical Reactions produced in a Solution: Vapour-tension of Solvent.—A. Ponsot.—The author finds that spontaneous and limited reactions, effected at constant temperature and pressure, between dissolved or mixed bodies, modify a homogeneous or heterogeneous system (1) by increasing, up to a maximum value, the vapour-tension of the solvent when it takes no part in the reaction; (2) by increasing, up to a maximum value, the vapour-tension of one of the reacting bodies when this body is produced in the reaction and inversely.

Estimation of, and Variation in, the amount of Cystine contained in Contaminated Waters.—H. Causse.—This paper is unsuitable for abstraction.

MISCELLANEOUS

Glasgow International Exhibition, 1901. — It is officially notified that all applications for space at the Glasgow International Exhibition, which is to be opened in May, 1901, must be lodged not later than the 1st of June with the General Manager, Mr. H. A. Hedley. There are in all eight classes, embracing agriculture, mining, industrial design and manufactures, machinery and labour-saving appliances in motion, locomotion and transport, marine engineering and ship-building, lighting and heating, science, education, music, sports and sporting appliances. Separate sections will be devoted to women's exhibits, archæology, and fine art.

Royal Institution.—The following are the Lecture Arrangements at the Royal Institution, after Easter:—Dr. Hugh Robert Mill, three lectures on "Studies in British Geography"; Dr. Alexander Hill, two lectures on "Brain Tissue considered as the Apparatus of Thought"; Mr. R. Warwick Bond, two lectures on (1) "Ruskin, Man and Prophet," (2) "Ruskin, the Servant of Art"; Professor Dewar, four lectures on "A Century of Chemistry in the Royal Institution"; The Rev. Canon Ainger, three lectures on "Chaucer"; Professor Stanley Lane-Poole, two lectures on "Egypt in the Middle Ages"; Dr. Alfred Hillier, two lectures on "South Africa, Past and Future"; Sir Frederick Bridge, three lectures on "The Growth of Chamber Music from Allegri's Symphonia (1580—1652) to Haydn's First Quartet" (with musical illustrations). The Friday Evening Meetings will be resumed on April 27th, when a Discourse will be given by The Right Hon. Lord Kelvin on "Nineteenth Century Clouds over the Dynamical Theory of Heat and Light"; succeeding Discourses will probably be given by Professor T. E. Thorpe, Mr. Sydney Lee, Professor J. A. Ewing, Mr. Francis Fox, Sir Henry Roscoe, and other gentlemen.

The Chemical Laboratory at Wiesbaden.—During the Winter Term of 1899-1900 the Chemical Laboratory Fresenius was attended by fifty-seven students. Of these forty-one were from Germany, five from England, two from Luxembourg, and one from Austria, France, Belgium, Italy, Spain, Russia, Sweden, Norway, and the United States of America. There were three assistants in the Instruction Laboratory, and twenty-four in the Versuchsstationen (private laboratories). In the teaching staff of the institution there has been no change. To this body belong the Directors, Professor Dr. H. Fresenius, Professor Dr. W. Fresenius, Professor Dr. E. Hintz, and also Dr. med. G. Frank, Dr. W. Lenz, Dr. L. Grünhut, and Architect J. Huber. The Summer Term begins on April 24th. In the Winter Term, 1899-1900, besides scientific work, there were many examinations carried on in the different departments of the private laboratory (Versuchsstationen) in the interest of trade, industry, mining, agriculture, hygiene, justice, and government.

MEETINGS FOR THE WEEK.

Wednesday, 18th.—Microscopical, 8. Demonstration on the Structure of some Palæozoic Plants, with Sections of the Plants thrown on the Screen, by Wm. Carruthers, F.R.S., President.

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NOTICE IS HEREBY GIVEN that EXAMINATIONS for the ASSOCIATESHIP (A.I.C.) and FELLOWSHIP (F.I.C.) will be held at the LABORATORIES of the INSTITUTE of CHEMISTRY, 30, Bloomsbury Square, London, W.C, on TUESDAY, July 10, 1900, and three following days.

The arrangements for the Examinations depend upon the number of Candidates. Candidates for the Final Examination may be required to present themselves from Tuesday, July 17, to Friday, July 20. Notice will be given to all Candidates whose applications for examination are received and accepted by the Council not later than Thursday, June 7, 1900, on which day the Examination Lists will be closed.

By Order of the Council,
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"The Regulations for Admission to Membership and Register of Fellows, Associates, and Students of the Institute of Chemistry" may be obtained from Messrs. Blundell, Taylor, and Co., 173, Upper Thames Street, London, E.C., price 1s.

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LECTURES.

Experimental Chemistry (Inorganic) — Prof. H. FRESENIUS, Ph.D.
Experimental Physics .. .. — Prof. W. FRESENIUS, Ph.D.
Stöchiometry .. ..
Organic Chemistry .. .. — L. GRÜNHUT, Ph.D.
Chemical Technology .. ..
Microscopy, with exercises in Microscopic work .. .. — W. LENZ, Ph.D.
Chemistry and Analysis of Foods .. — Prof. H. FRESENIUS, Ph.D., Prof. W. FRESENIUS, Ph.D., and Prof. E. HINTZ, Ph.D.
Hygiene .. .. — Dr. med. G. FRANK.
Practical exercises in Bacteriology .. — Dr. med. G. FRANK.
Technical Drawing, with exercises .. — J. HUBER.

The next Session commences on the 24th of April. The Regulations of the Laboratory and the Syllabus of Lectures will be forwarded gratis on application to C. W. Kreidel's Verlag, at Wiesbaden, or to one of the Directors.

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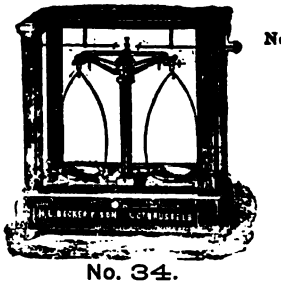

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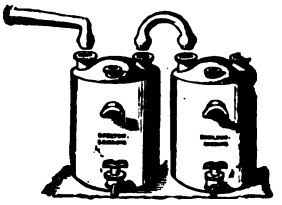

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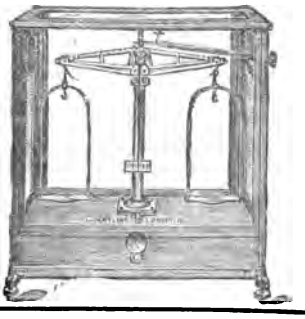

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THE CHEMICAL NEWS.

Vol. LXXXI., No. 2108.

A NEW METHOD OF SEPARATION OF THE GADOLINITE EARTHS AND PREPARATION OF PURE YTTRIA.*

By W. MUTHMANN and R. BÖHM.

(Concluded from p. 170).

Atomic Weight Determinations.

For the identification of Fractions V. and VI., which, by all their properties, must have consisted of pure yttria, equivalent weight determinations were carried out, the result of which we will briefly state. The final purification was carried out thus:—The oxides were heated for an hour over the blowpipe flame, then dissolved in the purest re-distilled nitric acid, and precipitated with oxalic acid; this operation was repeated four times in platinum vessels. Finally, the nitric acid solution was evaporated in a platinum crucible with pure sulphuric acid twice re-distilled from a platinum retort, and the excess of sulphuric acid removed by gentle heating on a sand-bath. The conversion of the sulphate into oxide is a slow process in the case of yttria; we had to heat for fifteen to twenty hours in the fiercest blowpipe flame before the weight became constant. The results were as follows:—

Fraction V.

	Grms.		Grms.
Sulphate..	1·75582	reduced to vacuum weights =	1·75636
Oxide ..	0·85570	" " "	0·85578
		SO_3 =	0·90058

Hence atomic weight = 90·12 (O = 16).

Fraction VI.

Sulphate	2·46505 grms.	reduced =	2·46585 grms.
Oxide	1·19511 "	"	1·19523 "
		SO_3 =	1·27062 "

Hence atomic weight = 88·97 (O = 16).

Cleve found for his yttria, most carefully purified by fractional precipitation of the oxalates, the atomic weight 89·02, which agrees so well with the number found by us for the sixth fraction that we do not hesitate to consider our preparation as absolutely pure yttria, while Fraction V. still contained small quantities of other earths with higher atomic weights.

Specific Weights.

The densities of the oxides afford an excellent means of tracing the advance of a fractionation in the case of the rare earths; and, in the yttria group, which contains earths of widely differing equivalent weights and widely differing specific weights, these important constants often make it possible to dispense with the lengthy equivalent weight determinations. When one considers that the density of yttria approximates to 5, that of erbium to 8·8, and that of ytterbium to 9·2, and that with careful work the pycnometrical method obscures complete accuracy of the determination in the second place of decimals, one sees at once that, with a mixture of two such earths, a density determination approximates to a quantitative method, but in both cases reliable conclusions are inferred as to the nature of the bodies under examination. We have therefore never omitted to determine these important constants in our work, and give in the following tables the numbers for the above-described fractions; in the last column the volumes of the oxides received from each fraction are added, whose sum obviously must be equal to the volume of the original material:—

Fraction.	Weight. Grms.	Density.	Volume. C.c.
I.	1·1	6·06	3·41
II.	19·6		
III.	7·4	5·62	1·31
IV.	7·3	5·43	1·34
V.	5·7	4·87	1·28
VI.	1·5	4·83	
	42·6		7·34
Original material	41·29	5·62	7·35

The correspondence is satisfactory, especially as the weights of the individual fractions were only determined approximately.

As is seen, the densities diminish rapidly from fraction to fraction, hence the earths with greatest equivalent weight are precipitated first.

Fraction III. shows the same specific gravity as the original material. It is remarkable that we found low numbers for pure yttria, viz., 4·83, while Cleve and Höglund give 5·028, and Nilson and Pettersson 5·046. On the other hand, a value 4·842, found by Ekeberg, agrees well with our numbers. The determination will be repeated later with more material.

Another very important fact results from our density determinations, viz., the presence of a considerable amount of an earth in the original mixture, whose specific gravity apparently lies between those of yttria and erbium. If it were assumed that it is principally these two oxides only that are present, the percentage for Yt_2O_3 would be 68·2, and for Er_2O_3 31·8, according to the simple equations—

$$x + y = 100$$

$$\frac{x}{4{\cdot}831} + \frac{y}{8{\cdot}640} = \frac{100}{5{\cdot}620};$$

where x and y are the required percentages.

But such a high percentage of erbium is not possible, for the spectroscope shows a maximum of 15 per cent, and the presence of another colourless earth is necessarily proved. Now it is to be remembered that we noticed three kinds of crystals, viz., red prisms, olive-coloured octahedra, and fine yellow needles. The first are identified as salts of yttrium, for pure yttria solutions yield only such prisms. We consider the octahedra to be rich in erbium, for they occur principally in the first fractions, which show an intense erbium spectrum, and hence the third oxide must be concentrated in the yellow needles. An experiment carried out with more material—about 2 kilogrms.—which one of us has started in conjunction with Postius, will settle this point. We might mention that lanthanum is quite excluded, as it gives with potassium chromate a quite characteristic, apparently amorphous precipitate, which in reality consists of fine crystalline globules. Also the remaining cerite earths, such as thorium, samarium, and gadolinium, do not come into consideration, so that only terbium and ytterbium remain. It is certain that the quantity of yttria in the gadolinite oxide mixtures is less than has hitherto been assumed.

In conclusion, we should like to indicate how unusually quickly the methods we have discovered can be carried out for fractionating earth mixtures. We succeeded in less than a week to obtain from the already described material about 15 per cent yttria in the absolutely pure state. This must be looked upon as a very fair result, for the earlier used methods are very tedious, and for this reason

* *Berichte Deutsch. Chem. Gesell.*, Part 33, No. 1.

Cleve is the only one who has hitherto succeeded in obtaining pure yttria; Bahr and Bunsen only arrived at getting an earth of atomic weight 92·63. The remaining rare earths, especially those of the cerite group, can be relatively easily separated by our methods; we shall in a short time publish our results for this group. We may already remark that we have easily succeeded in separating samarium from the monazite oxides.

It naturally requires more exhaustive and laborious experimenting to discover the most favourable conditions for a separation. The idea of precipitating the rare earths fractionally as chromates is not really new, as Krüss and Loose have already studied the influence of potassium chromate on solutions of the nitrates. But they came to no real conclusion, and a repetition of their attempts only showed that it was not a possible means, for, instead of pure chromates, mixtures of these with basic nitrates were produced in amorphous form, and therefore the experience of Krüss and Loose affords no more than fractional precipitation with ammonia.

Inorganic Chemical Laboratory
of the Technical High School, Münich.

ELECTROLYTIC METHOD FOR THE PREPARATION OF ALKALINE CHLORATES, BROMATES, AND IODATES.

By M. MULLER.

Förster and Jorre (*Journ. f. Prakt. Chem.*, 1899, lix., p. 53) have recently shown that the electrolytic formation of alkaline chlorates is due to the oxidation of the hypochlorite from the chloride by free hypochlorous acid. Having obtained this result, the question of the influence of other oxidising agents naturally comes forward.

The following table shows with what rapidity equal quantities of acetic, sulphuric, and chromic acids added to a solution of hypochlorite transforms this salt into chlorate.

In this experiment, as in the following ones, the hypochlorite was estimated by Penot's method, the total oxygen by iodometry, and the oxygen in the state of chlorate by difference, following the directions given by Förster and Jorre.

During the action of acids on hypochlorite of potash, the chlorate commences to crystallise after about one hour. A special sample must be taken for the estimation of the total oxygen.

One c.c. of acid is allowed to react on 1 c.c. of the hypochlorite solution, and after seventeen hours the total oxygen in the mixture is estimated. In the case of chromic acid, the oxygen corresponding to the chromic acid is subtracted from the total oxygen, for this acid would also take part in the reaction during the iodometric estimation, as we must admit that its quantity has not varied.

Experiment I.

Time lapsed since mixing.	10 c.c. of hypochlorite solution were added to—			
	10 c.c. water.	13 c.c. normal acetic acid.	10 c.c. normal sulphuric acid.	10 c.c. normal chromic acid.
	Grms. of oxygen as hypochlorite in 100 c.c.			
—	1·45	—	—	—
10 minutes ..	—	0·96	0·92	0·88
35 „ ..	—	0·62	0·62	0·42
60 „ ..	—	0·55	0·56	0·32
2 hours.. ..	1·45	0·49	0·55	0·26
4 „	—	0·41	0·49	0·16
17 „	1·44	0·30	0·29	0·10
	Total grms. of oxygen in 100 c.c.			
At commencement	1·52	—	—	—
After 17 hours.	1·52	1·48	1·46	1·49

Since the transformation of hypochlorite into chlorate is greatly accelerated in the presence of chromic acid, one might suppose that by adding chromate of potassium to a solution of chloride of potassium and electrolysing without a diaphragm, the hypochlorite produced would be oxidised into chlorate more rapidly, and that the losses due to the reduction caused by the current would be thus diminished.

To examine the influence of the chromate, two experiments were made according to the instructions given by Œttel (*Zeit. f. Elekt.*, 1895, Nos. 11 and 15).

A glass vessel was used, of 550 c.c. capacity. A strip of platinum of about 29 square c.m. served as the cathode, on each side of which, at a distance of about 1·5 c.m., was a much smaller anode, also of platinum. The electrodes, and a glass tube for the escape of gas, were fixed through the cork which hermetically sealed the vessel. A gas voltameter, a copper voltameter, an ampère-meter, and a voltmeter were placed in the circuit. During the electrolysis several samples of gas were taken. The results are given in the following table. The strength of the current was 4·5 ampères.

Experiment II.—*The Solution contained* 30 *per Cent of* NaCl.

Temperature, 45—50°. Tension, 3·9 to 4 volts.
D_c = 0·18 amp./sq. c.m. D_a = 0·075 amp./sq. c.m.

Time. Hours.	Return from current. Per cent.	Reduction. Per cent.	Decomposition of water. Per cent.
1	52·3	41·3	6·4
2	35·5	53·3	11·2
3	33·4	54·7	11·9
4	33·1	54·5	12·4
18	30·8	56·7	12·5
21	32·8	54·8	12·4

There were 112 grms. of copper precipitated in the voltameter, corresponding to 28·2 grms. of O_2. The solution contained 9·26 grms. of active oxygen, or 32·1 per cent *of the theoretical return.*

Experiment III.—*The Solution contained* 30 *per Cent of* NaCl *and* 0·18 *per Cent of* K_2CrO_4.

Temperature, 42—50°. Tension, 4·1 to 4·7 volts.
D_c = 0·18 amp./sq. c.m. D_a = 0·075 amp./sq. c.m.

Time. Hours.	Return from current. Per cent.	Reduction. Per cent.	Decomposition of water. Per cent.
1	83·5	3·8	12·7
2	77·6	2·4	20·0
3	69·5	0·8	29·7
4	71·6	2·1	26·3
18	68·7	2·8	28·5
21	67·9	3·6	28·5

There were 102·05 grms. of copper deposited in the voltameter, corresponding to 25·75 grms. of oxygen. The solution contained 17·92 grms. of active oxygen, or 69·6 per cent *of the theoretical return.*

An experiment made under the same conditions as the last, but at 10° for twenty-three hours, gave a still better return; perchlorate was also formed in a noticeable quantity. This might be used as a method for preparing perchlorates directly from chlorides.

The return of 69·6 per cent obtained in Experiment III. surpasses, not only anything that was done in neutral solution, but even the best results obtained by Œttel in alkaline solution.*

It can be seen that this result is obtained from the fact that the reduction is greatly diminished; it therefore seems, at first sight, to agree with the suggestion we have already made. The estimation of the quantity of hypo-

* This return is not the highest possible. The author will shortly describe a process by means of which 88 per cent of the theoretical return can be obtained.

chlorite in solution will, however, show that in this case it is not right to form a conclusion with regard to the proportion of this salt from the reduction figures obtained by the analysis of the gases.

The amount of hypochlorite in 500 c.c. of solution was, at the end of Experiment II., 1·15 grms.; at the end of Experiment III., 1·79 grms.; and the last reduction figures obtained were 54·5 per cent and 3·6 per cent. We therefore see that the highest value of the reduction corresponds with the lowest quantity of hypochlorite. We must therefore conclude that, in some way or another, the action of the chromate retards the reduction at the cathode.

It thus appeared to be possible that an addition of chromate would also favour the production of bromates and iodates by electrolysis. As we already know (*Zeit. f. Elekt.*, 1897, p. 474; *Journ. f. Prakt. Chemie*, 1897, lvi., 353) those salts are again reduced by the current to the state of bromides and iodides in such a way that after a little while the return becomes very low. The preceding supposition is, in fact, found to be perfectly true. Without wishing to here record all our unsuccessful experiments, we will quote two carried on for a long time under conditions that we considered to be very favourable.

The experiments were carried out in a glass vessel of 120 c.c. capacity, closed like the preceding one. Two strips of platinum of 10 sq. c.m. area, about 1 c.m. apart, served as electrodes. The intensity of the current was 1 ampère.

EXPERIMENT IV.—*The Solution* (100 *c.c.*) *contained* 20 *grms.* KI, 1 *grm.* KOH, *and* 0·18 *grm*, K_2CrO_4.

Temperature, 40°. $D_a = D_c = 0{\cdot}1$ amp./sq. c.m.
Tension = 3·3 to 3·7 volts.

Time. Hours.	Return from current. Per cent.	Reduction. Per cent.	Decomposition of water. Per cent.
1	98·7	9·8	0·5
2	97·5	1·4	1·1
4	99·1	0·9	0·0
15	96·7	0·0	3·3
16	91·0	1·8	7·2
17	91·4	1·3	7·3
19	81·7	0·4	17·9
21	27·3	0·6	72·1
22	9·1	4·3	86·8

There were deposited 26·18 grms. of copper in the voltameter, corresponding to 6·6 grms. O_2. By crystallisation we obtained 21·5 grms. of KIO_3, corresponding to 4·823 grms. O_2, and the solution still contained 0·8258 grm. O_2, or a total of 5·6488 grms.; that is to say, 85·6 per cent of the theoretical return. If we neglect the two last hours, we obtain 97 per cent *of the theoretical return*. Theoretically, 20 grms. KI should be entirely transformed into KIO_3 at the end of 19·3 hours. We obtained 97·7 per cent of iodide oxidised to iodate.

EXPERIMENT V.—*The Solution contained* 20 *per Cent of* KBr *and* 0·18 *per Cent of* K_2CrO_4.

Temperature = 32—36°. $D_a = D_c = 0{\cdot}1$ amp./sq. c.m.
Tension = 2·5—4·1 volts.

Time. Hours.	Return from current. Per cent.	Reduction. Per cent.	Decomposition of water. Per cent.
½	97·3	2·1	0·6
3	94·8	2·5	2·7
13	93·7	4·1	2·2
14	90·6	6·1	3·5
19	93·1	3·7	3·2
21	94·5	3·3	2·2
23	90·7	4·4	4·9
25	90·2	3·9	5·9
26	91·8	3·4	4·8
28	82·5	3·1	4·4
30	64·2	3·1	2·7

In the voltameter were deposited 34·75 grms. of copper, corresponding to 8·77 grms. of oxygen. On crystallisation we obtained 19·85 grms. of $KBrO_3$, corresponding to 0·706 grm. of O_2. There remained in solution 2·1752 grms. of O_2. This makes a total of 7·8812 grms. O_2, or 89 per cent of the theoretical return. In this case, again, the return from the first twenty-one hours gives a mean of 94 per cent *of the theoretical return*. Theoretically, the 20 grms. of Br ought to have been oxidised into $KBrO_3$ after 26·9 hours. We obtained 37·6 per cent of the bromide oxidised to bromate.

We obtained just as good results with soda salts, and on a much larger scale, using 500 c.c. of a 20 per cent solution. We also made a certain number of experiments to determine the most favourable conditions from several different points of view.

To obtain a pure product it is necessary to use a platinum anode; with a carbon anode the solution becomes coloured brown, as also are the crystals which are formed. Powdered carbon is also deposited; this is removed mechanically from the electrodes—the salts crystallising on the surface of the carbon appear to disintegrate it. As a cathode, we may use platinum, iron, nickel, or copper; lead and zinc are not suitable. The following figures give the reduction in the electrolysis of a neutral solution of NaBr with a cathode formed of these metals:—

EXPERIMENT VI.

Metal of the cathode.	Reduction. Per cent. Maximum.	Minimum.
Platinum	0·2	2·9
Nickel	2·1	2·5
Iron	2·6	3·9
Copper	2·9	3·8
Lead	5·6	15·5
Zinc	20·0	30·8

As for the composition of the electrolyte, an addition of an alkali increases the quantity of water decomposed in the case of bromides and chlorides, but it has no influence on the return with iodides; this was to be expected in view of the values of the potential of discharge of the ions of hydroxyl and of the halogens.

It is important, at least with iodides and bromides, to continually stir the electrolyte, or otherwise the salt in crystallising will entangle some of the halogen.

Concentrated or dilute solutions are equally good for electrolysis. In fact, we only notice a diminution in the return when the solution is very dilute indeed.

We also tried making the proportion of chromate variable. If it is increased the decomposition of water increases slowly, if it is diminished it is the reduction that is increased.

The salts in which the chromium is in the state of oxide play the same part as the chromate; they are oxidised by the current. In any case, at the end of the experiment, we do not find that the proportion of chromic acid has varied.

As a general rule the experiments are carried on at a high temperature, so as to diminish the voltage. With chlorides, it must be remembered that below 25° we obtain a notable proportion of perchlorate. With iodides and bromides the return is equally good, hot or cold, as neither the bromates nor the iodates can be oxidised by electrolysis, even at a very low temperature. In such a case the temperature should not exceed 50°, as at a higher temperature the reduction is stronger.

The density of the current may be varied between wide limits, as shown in the experiments. It is difficult to explain the influence of the chromate.

Whatever it may be, it is noticed that after the experiment the cathode has a peculiar appearance, which is not that of clean platinum; this is caused by the chromium. There are two possible cases; it may be that the cathode becomes covered with a thin layer of chromic hydrate, which acts like the lime which forms a diaphragm interfering with the reduction, in the electrolysis of solutions

of chloride of calcium (*Zeits. f. Elektr.*, 1898, No. 20). This would explain why the voltage was higher for solutions containing chromate, although this might also be explained by a variation in the polarisation of the hydrogen. But the fact that the hypochlorite rapidly oxidises the chromic hydrate into chromic acid is contrary to this hypothesis; it would then be necessary for the hydrogen to reduce CrO_3 to Cr_2O_3, in the same way that NaOCl oxidises Cr_2O_3 to CrO_3, and in this case the value of the reduction would not give any indication.

It might also be possible for an alloy of chromium to be formed on the surface of the cathode. It is an experimental fact (*Zeits. f. Elektr.*, 1896, No. 16) that the alkaline chlorates undergo reduction, more or less, according to the metal used as cathode, and it may be imagined that the metallic chromium formed on the surface of the cathode would interfere with reduction. As a matter of fact, the metals which interfere with the reduction have a close, dead-white appearance at the end of the electrolysis, while zinc and lead become covered with an easily-removed porous material. This phenomenon might perhaps be cleared up, if we admit that all the metals which form a porous non-compact alloy with chromium during electrolysis are unable to prevent reduction.* We may here mention that the salts of manganese have not the same action.

Finally we would remark that, to all appearance, an addition of chromate is an advantage in every case when the products of oxidation at the anode require to be protected against reduction. — *Zeitschrift für Elektrochemie*, v., p. 469.

THE

DETERMINATION OF ANTIMONY IN ORES.†

By THOMAS BROWN, Jun.

(Concluded from p. 179).

1. *The Soluble Ores.*—For the "*soluble ores*" 1 grm., which has been finely pulverised in an agate mortar, is treated in a No. 3 beaker with 25 c.c. concentrated hydrochloric acid, covered and boiled on a plate or over a flame until apparently decomposed and the solution is not more than 15 c.c. in bulk. About 2 grms. of tartaric acid are added, and as soon as that is dissolved about 4 to 6 drops of concentrated nitric acid, and the whole is boiled for a minute or so more. This last addition causes a violent reaction and a more complete decomposition. The beaker is now removed from the source of heat, the cover and sides washed down, the insoluble residue filtered out, and the filtrate and washings diluted in bulk to about 250 c.c. A steady stream of hydrogen sulphide gas is now run through the solution for about an hour until it is thoroughly saturated and the antimony thus precipitated as the amorphous orange-coloured sulphide (Sb_2S_3).

To insure a complete precipitation this is gently warmed in order to convert any basic chloride into sulphide, and then allowed to stand an hour or so in the cold before filtering. The filtrate will then be perfectly clear, and not deposit additional antimony upon standing or upon further treatment with hydrogen sulphide gas. Should lead or other impurities of the fifth group have been found by the preliminary qualitative tests, after filtering and washing the precipitate with cold water it is washed back into the beaker in which it was precipitated, and treated with ammonium sulphide in such excess that with the aid of heat the antimony precipitate completely dissolves. The solution is then run back through the same filter, caught in a clean beaker, and the filter thoroughly washed with hot water. The solution and washings are acidified in slight excess with hydrochloric acid, re-precipitating the antimony sulphide, diluted to about the same bulk as before (250 c.c.), allowed to stand a few minutes to settle, and then filtered. In the black sulphide residue remaining on the filter representing the impurities of the fifth group, the lead may now be determined by placing the filter containing this precipitate into a beaker, treating with nitric acid and boiling, adding ammonia in excess, then acetic acid in excess, filtering, re-precipitating the lead as chromate, and weighing it thus on a Gooch crucible or balanced filter-papers.

The antimony sulphide precipitate, whether precipitated but once or re-precipitated for purification, is now washed well upon a large, plaited, balanced filter, with cold water, and dried in an air-bath at 110° C. for two hours or more to constant weight. After removal, and as soon as cold, the weight of the dried precipitate is taken, which consists of antimonous sulphide (Sb_2S_3) mixed with a small amount of free sulphur.

The following procedure has been found both rapid and accurate for weighing this precipitate. A German corrugated filter, 15 c.m. in diameter, is used. Two are selected which lay beside one another in the same pack, and which from long impact contain practically the same amount of moisture. They are balanced by cutting down the heavier one. Upon the unaltered one the precipitate is filtered, the other accompanying it in the drying oven and as a counterpoise in weighing. When the weight of the precipitate has thus been determined, it is carefully scraped from the paper and powdered, and mixed well by grinding in a small glass mortar. A certain amount of this (5/10ths grm.) is weighed off into a platinum boat, this introduced into a glass combustion-tube, and the free sulphur driven off in a stream of carbon dioxide. From the weight of the resulting black anhydrous antimonous sulphide so obtained on a weighed amount of the dried precipitate, its total weight thus treated can easily be calculated, and it is then simply a matter of calculation to determine the total amount of antimony present. $Sb_2S_3 \times 0{\cdot}7177 = Sb$.

The combustion requires in all from twelve to fifteen minutes, and, if carefully conducted with the following precaution, very little loss if any at all occurs. Arsenic if present would be volatilised. (*a*). A steady stream of gas should be passing through the tube when the flame is applied underneath the boat, and should continue so with as little variation as possible until the end of the operation. Should this not be the case, a flame would at first play over the surface of the contents of the boat, causing oxidation apparent to the eye when removed from the tube by a whitening of the surface and consequently a loss in weight. (*b*). The heating of the boat in the tube should be accomplished gradually. At first a gentle heat is applied until all the sulphur has apparently been driven off, and increased towards the end of the operation. (*c*). The tube should be cleaned of sublimed sulphur by heating before each and every determination. After weighing the boat and contents it is well to treat again for additional loss in weight, which is rarely found if the operation has been conducted as described.

2. *The Practically Insoluble Ores.*—The important point here is the decomposition of the ore which is accomplished by fusion. One grm. of the finely-powdered ore is mixed with 8 to 10 parts of a flux composed of equal parts of flowers of sulphur and sodium carbonate, introduced into a porcelain crucible (capacity 40 c.c.), and in addition the mixture is covered with a thin layer of the flux. The covered crucible is introduced into the opening of a muffle that is of a bright red heat, and is cautiously heated to fusion for about ten minutes. The heat applied at first should be of such an intensity that sulphur flames are barely seen escaping around the edge of the lid; after they cease it is somewhat increased by pushing the crucible a few inches farther back in the muffle. At no time should the crucible become heated to more than per-

* Possibly these facts are in accordance with those observed by Brœlig and Haber (*Deutsch. Ch. Ges.*, 1898, xv., p. 2741).

† From the *Journal of the American Chemical Society*, vol. xxi., No. 9.

ceptible incandescence on the side facing the source of heat, and during the operation it should be occasionally turned with a pair of tongs to insure an even heating and consequently a complete decomposition. The colour of the melt when cold should be green, not yellow, and likewise the solution of the same, due to the sulphide of iron present. A suggestion of a yellow colour particularly in the solution indicates an incomplete decomposition of the ore, and but a partial change of the antimony into the soluble sodium sulphantimonate, which consequently necessitates a repetition of the fusion.

A successful fusion, owing to the ready volatility of antimony at a comparatively low degree of heat, is rather a delicate thing and difficult to accomplish, requiring constant care and judgment only gained by experience. With the statement that it is possible to thoroughly decompose such an ore by fusing over a candle-flame, the writer is compelled to differ, having frequently found that a fusion made at a seemingly alarming degree of heat gave, when dissolved, a yellow solution in which the antimony when determined was found to be lower than that of a duplicate sample which had been treated to a higher degree of heat. It is, therefore, better to treat in excess than not to fuse enough; for should there be a slight loss by volatilisation, this would in a measure be counterbalanced by the small amount of impurities, such as silica, obtained from the action of the flux on the crucible and on the silica of the ore, and consequently thrown down by the acid and weighed with the antimony sulphide precipitate. The degree of heat used, however, should scarcely affect the glaze of the crucible. When the fusion has thus been satisfactorily accomplished, and when cool, it is dissolved in hot water, which takes but a few minutes; the suspended precipitate of iron sulphide and sulphides of the metals of the fourth group, if present, filtered off, and the antimony precipitated by adding a slight excess of hydrochloric acid to the filtrate. After stirring and allowing to stand a few minutes to settle, the precipitated antimonic sulphide (Sb_2S_5), mixed with more or less free sulphur, is filtered off upon a balanced filter, and so determined as before, being ultimately weighed when removed from the combustion-tube as antimonous sulphide (Sb_2O_3).

3. *The Partially Soluble Ores.*—These ores require a double treatment, that is, they are first treated as for soluble ores and the antimony determined in the acid solution, and the residue—remaining on the filter which contains the undissolved oxide—is subjected to a fusion as for oxide ores. This filter-paper is placed in the bottom of a porcelain crucible and covered well with flux, so that some comes in direct contact with its contents. It is then fused as directed. In dissolving, pieces of charred paper may possibly be found, which are, however, filtered out together with the insoluble matter. The amount of antimony so present is determined as before.

Most Mexican so-called sulphide ores exported are really oxysulphides, being mixtures of sulphide with a small amount of oxide which has not been separated in the sorting: these ores consequently require this double treatment. The following is a list of results obtained in using these methods upon the previously mentioned varieties of Mexican ores, and gives in the case of oxysulphides the soluble and insoluble antimony found. In the last column will also be found the results by the fire assay, which is extensively used on oxide ores for prospecting work, and also for large shipments when the "wet assay" is to be made later at their destination. It is figured by the dealers that the wet assay of a 60 per cent ore will go 6 per cent higher than the dry or fire assay. That there are exceptions to this will be seen in Nos. 5, 8, and 12 of the table. No. 5 is the senarmontite ore, the complete analysis of which has already been given, while Nos. 8 and 12 are quite insoluble under the acid treatment, leading to the supposition that they are cervantite ores of the composition Sb_2O_4. It has also been observed that certain grades of oxide ores give higher results by fire assay than other grades, which is considered due to their ultimate composition, of which very little is so far known. The widely different and extremely low results obtained by fire assay upon sulphide ores makes them no criterion upon which to judge of the actual antimony present, and so for this class of ores it is falling into disuse.

No.	Class of ore.	Soluble Sb.	Insol. Sb.	Total Sb.	Fire assay.
1.	Sulphide ..	67·4	—	67·4	—
2.	,, ..	28·5	—	28·5	—
3.	Oxysulphide .	61·6	5·1	66·7	48·0
4.	,, ..	63·3	6·7	70·0	45·0
5.	Oxide	70·7	3·1	73·8	72·8
6.	,, ...	—	—	71·7	65·0
7.	,, ...	—	—	66·1	59·0
8.	,, ...	—	—	63·9	61·8
9.	,, ...	—	—	55·8	49·0
10.	,, ...	—	—	49·0	43·0
11.	,, ...	—	—	48·3	43·0
12.	,, ...	—	—	46·4	44·4

The fire assay referred to consists of the reduction of the contents of the ore to the metallic state by fusion with potassium cyanide, and while it is known that it is possible to obtain higher results by the use of other mixed fluxes together with a treatment for a longer time at a lower heat, this cyanide reduction remains the accepted method in trade. It is performed as follows:—Five grms. of the powdered ore is mixed with 12 parts of potassium cyanide, placed in a 10 grm. clay crucible, and covered with salt. The fusion is made in a muffle at a bright red heat, allowing it to remain there thirteen minutes. When removed it is thoroughly shaken to collect the globules into one metallic button, and allowed to cool. The crucible is broken, and the resulting button carefully broken from the slag and weighed.

In conclusion the writer desires to express his obligation both to Mr. M. Eisasser and to Mr. Ferdinand McCann, for their kind assistance in furnishing him with suggestions, data, &c., without which this paper would not have been written.

ON THE DETERMINATION OF SULPHURIC ACID IN THE PRESENCE OF IRON:

A NOTE UPON

SOLID SOLUTIONS, AND THE HYDROLYSIS OF CHROMIC AND FERRIC SALTS.*

By THEODORE WILLIAM RICHARDS.

Within the past year appeared three interesting articles by Küster and Thiel (*Z. Anorg. Chem.*, 1899, xix., 97; 1899, xxi., 73; 1900, xxii., 424), concerning several methods of precipitating baric sulphate free from iron when that element has been present in the solution. One of the methods which they have proposed is an excellent addition to our always imperfect analytical repertory, and the chemical world owes them a debt for the ingenious although exceedingly simple suggestion.

While not wishing in the least to detract from the merit and interest of this work, I feel that attention should be called to several omissions in their paper, especially those which concern theoretical considerations involved in previous work upon the subject.

In their first paper, where the admirable analytical device is explained, Küster and Thiel ascribe the "occlusion," of iron to the presence of the ferric ion. It is interesting that this idea, which they themselves have since shown to be false, should have led them to the

* Contributions from the Chemical Laboratory of Harvard College. A part of this paper appeared in *Proc. Amer. Acad.*, 1900 xxv., 377, and it is published in full in *Zeit. Anorg. Chem.*

desired goal. The circumstance might be taken as an argument in favour of the proposition that even an incorrect idea is better than none. In a reference to this paper, Ostwald (*Z. Phys. Chem.*, 1899, xxix., 340), points out that the occlusion is in all probability due not to the ferric ion, but rather to a molecular complex; and in their last paper, Küster and Thiel describe experiments which prove beyond much doubt the correctness of Ostwald's suggestion. Oddly enough, however, they still cling, in their summing up of the matter, to the statement that the ferric ion is the essential agent.

It is curious also that they should have overlooked the following statement in a paper published over ten years ago by Professor Jannasch and myself. In speaking of the cause of the phenomenon under consideration, we said:—"Es lässt sich unmittelbar annehmen, dass das Eisen als eine Doppelverbindung mit Baryum als Baryum-Ferrisulphat gefällt wird, welches wahrscheinlich ein Molekül gebundenes Wasser (sogen. Constitutionswasser) enthält" (Jannasch and Richards, *Journ. Prakt. Chem.*, 1889, [2], xxxix., 321). Küster and Thiel have offered new arguments which they consider as being in favour of the first part of this incomplete proposition, without appearing to recognise that the proposition is not itself new. They have moreover ascribed an arbitrary formula to the substance, while we did not feel prepared either to assume its exact nature or to explain the mechanism of its occlusion.

Three years after this publication Schneider described some interesting experiments in which evidence was given that the occlusion of the double or complex compound is a variety of solid solution (E. A. Schneider, *Zeit. Phys. Chem.*, 1892, x., 425). The idea was new at the time, and did not meet with general favour; but a knowledge of the work would have saved Küster and Thiel trouble.

In 1894 (T. S. Gladding, *Journ. Amer. Chem. Soc.*, 1894, xvi., 398; 1895, xvii., 397), after the passing of two more years, Gladding published a modification of Lunge's method of precipitating baric sulphate free from iron, which is the most convenient of any which had appeared up to that time. He precipitated the iron with ammonia, filtered without especial washing, acidified the filtrate, added an excess of baric chloride, and then collected on one filter both the main mass of the baric sulphate and the small amount of the same substance which he obtained from the ferric hydroxide. The chief difference between this method and the method of Küster and Thiel is the fact that the latter pointed out the uselessness of filtering off the ferric hydroxide before adding the baric chloride. It is quite probable, however, that Küster and Thiel did not know of Gladding's work, and hence independently devised the whole process.

Let us consider for a moment the nature and cause of this kind of occlusion. In the first place it must be distinguished sharply from the mechanical retention of mother-liquor in minute cells which all crystalline precipitates exhibit. Even large crystals rarely contain cells of included mother-liquor plentiful enough to increase their weight by more than the fraction of a per cent; and this impurity is of course chiefly water. It is easy to test the magnitude of this *inclusion* by precipitating such a substance as calcic carbonate from a solution containing a large quantity of some easily identified substance such as sodic chloride. Mr. R. P. Cushing kindly made a number of such experiments for me, and found that not even from a saturated solution of salt did more than 0·1 per cent of the precipitate consist of sodic chloride. Of course this value fixes the maximum, for some of the substance may well have been *occluded* instead of *included*.

Very different from the mechanical process of *inclusion* is the behaviour of baric sulphate. Here the impurities may amount to several per cent, and the foreign material is disseminated equally throughout the mass. The experiments of Schneider show that the amount of the occlusion is greater as the concentration of the impurity in the solution increases, although not in direct proportion. It is very clear that we are dealing here with a special case of the distribution law; Küster and Thiel evidently thought of this possibility, but they did not amplify the idea. The distribution can take place only at the moment of precipitation, because afterwards the rigidity of the solid prevents free motion. In the present paper I have retained the name occlusion for this phenomenon because no other term seems to be applicable. There is indeed a certain analogy between the occlusion of hydrogen in palladium and the dissemination of a solute out of a solution into a solid; and since the name gives rise to no misunderstanding it will answer its purpose.

Obviously the study of the distribution law in other cases should throw light upon this one by analogy. When hydrochloric acid is distributed between its aqueous solution and the vapour phase, the concentration of the undissociated part alone is concerned in the distribution, because ions can exist only in the solution. In the same way, when a moderately strong acid is distributed between water and ether, only the undissociated part of the acid comes into play, because ether does not cause important dissociation. From these analogies one might infer that the group which is concerned in cases of concomitant precipitation are not ions, but rather electrically neutral complexes. The inference is not a perfectly safe one, for little is known about the possible degree of dissociation in solids: but experimental evidence is at hand to support this view. In 1894 Richards and Parker made the observation that the occlusion of baric chloride by baric sulphate is much increased by the previous addition of hydrochloric acid to the solution of baric chloride (*Proc. Amer. Acad.*, 1894, xxxi., 74; *Zeit. Anorg. Chem.*, 1895, viii., 420). For example, the addition of 20 c.c. of strong hydrochloric acid caused almost twice as much occlusion as the addition of 10 c.c. in another otherwise similar case. The conditions attendant upon mixing two liquids are so uncertain that it is unsafe to attempt the quantitative interpretation of the relationship; but qualitatively this result is wholly in accord with the law of mass action, provided that the undissociated group is supposed to be the one concerned.

The nature of the electrically neutral group with which we have to do in the present case is less easy to imagine. Küster and Thiel assume that it is $Ba[Fe(SO_4)_2]_2$; but this is evidently an unsafe assumption. The analogy to the case of chromium which they quote is an excellent point, but they apply their useful analogy in an incomplete fashion.

Recoura (*Amer. Chem. Phys.*, [4], vii., 497), and Whitney (*Zeit. Phys. Chem.*, 1896, xx., 40), and others have shown that a "modified" green solution of chromic sulphate certainly contains an acid. In other words, it is hydrolysed, and must likewise contain a basic complex. It has also been shown that in the presence of *much free sulphuric* acid, a complex of an opposite nature, chromosulphuric acid, $H_2SO_4.Cr_2(SO_4)_3$, is formed. None of these investigators have completed the explanation by the following hypothesis, however.

As the acid is set free by the progressing hydrolysis of the normal violet chromic salt, this acid must simultaneously cause the formation of chromosulphuric acid. In short, a reaction somewhat like the following must take place:—

$$Cr_2(SO_4)_3 + H_2O \rightleftarrows CrSO_4OH + HCr(SO_4)_2.$$

Perhaps this statement does not exactly represent the proportions; but nevertheless it accords with the quantitative results of Whitney and Recoura. It will be remembered that they found each molecule of chromic salt capable of yielding one replaceable hydrogen, and also that only a third of the sulphate was capable of immediate precipitation. This explanation is much simpler than the complicated assumptions of Recoura.

According to our first hypothesis that compound which is most dissociated should be least occluded by baric sulphate. Now the conductivity measurements of Whitney

show that the acid complex is far more dissociated than the basic one; hence I am inclined to ascribe the occlusion chiefly to the basic substance. This interpretation is diametrically opposite to the interpretation of Küster and Thiel.

Nevertheless, among the results of Küster and Thiel's work, we find other arguments indicating the basic nature of the occluded body. On pages 439—440 of their third paper they point out that the addition of acid diminishes very much the occlusion of the iron complex. This is wholly consistent with the present thesis: for (according to the Mass Law) the addition of acid must diminish the concentration of the basic complex in the solution, and hence (according to the distribution law) the amount which finds its way into the solid. It is true that this argument is somewhat diminished in force by two circumstances: first, because Küster and Thiel have not here considered the fact that hydrochloric acid increases the weight of the precipitate by introducing into it more baric chloride; and second, because the addition of hydrochloric acid undoubtedly causes a chloride-complex which is not occluded to nearly so great an extent as the sulphate-complex by the baric sulphate. In order really to study the effect of the hydrogen ion one should not rely simply upon the total weight of the mixed precipitate, but should actually determine the amount of iron present in the precipitate and compare it with the amount present in a precipitate obtained from a solution containing the same amount of iron and an amount of sodic chloride equivalent to the hydrochloric acid used in the first case. In spite of these objections, however, the effect observed by Küster and Thiel is so great that one must ascribe a portion of it to the action of the hydrogen ion, and hence, according to Whitney and Recoura, infer the substance concerned to be a basic complex. Küster and Thiel offered no explanation for the action of the hydrogen ion.

Again, Küster and Thiel point out (pp. 437—438) that the hydrolysis caused by dilution prevents the addition of much water from causing as considerable a decrease in the absorbed material as would otherwise have been expected. This is equivalent to saying that the complex which is concerned is a basic one; for the hydrolysis of course involves the ionisation of hydrogen.

Yet another, although less cogent, argument in favour of the basic nature of the complex is the fact that Jannasch and I found the impure baric sulphate to be capable of retaining about 3 per cent of water at 250°. While it is not impossible that this should be at least in part simply held in minute cells or as crystal-water, one is inclined to ascribe part of it to hydroxyl existing in the precipitate. It may well be, however, that the extra water is simply dissolved in the precipitate, as other undissociated substances are dissolved by baric sulphate. This tendency to dissolve water may be the reason why many precipitates require prolonged ignition in order to attain constant weight.

Over a year ago I was attracted by the well known occlusion of green chromic sulphate by baric sulphate, into beginning a study of the subject of occlusion with the help of this phenomenon. Chromium was selected instead of iron because its complex is more stable and has been more thoroughly studied. The research was begun by Mr. R. P. Cushing, and is being continued by Mr. F. Bonnet. Since the precipitated baric sulphate is green, one must infer immediately that the green modification (or complex) is responsible for the occlusion. The work is not yet ready for publication, but our data agree well with those of Küster and Thiel, while our explanation accords with that given in the present paper. We are also studying aluminum sulphate, which likewise seems to form a complex very considerably occluded by baric sulphate; although this fact does not seem to be generally known (compare Peckham, *Journ. Amer. Chem. Soc.*, 1899, xxi., 772).

This paper is intended to serve as an introduction to two other papers upon specific cases of occlusion. The chief points upon which emphasis is desired are the following:—

1. *Occlusion* from solutions differs from *inclusion* by being a more intimate and a less mechanical mixture.

2. Occlusion differs from *adsorption* in concerning the whole mass of the precipitate, and not merely its surface. Hence after a precipitate is once formed occlusion takes place only with extreme slowness, because of the rigidity of solids. New evidence of this will be given in one of the following papers.

3. Different precipitates have almost as widely varying powers of occluding other substances as different liquids have widely varying powers of dissolving other substances. The property is no more to be predicted than the power of dissolving.

4. The process of occlusion is probably the distribution of an electrically neutral simple substance or complex between the solution itself and the solid at the moment of its formation within the solution. Hence any change in the substances present in the solution which tends to diminish the concentration in the solution of the group under consideration, tends to diminish the amount occluded.

5. The nature of the groups which are occluded in the cases of iron, chromium, and aluminum is not yet determined, but they are probably basic in nature, and identical with the complexes of Recoura and Whitney.

6. Green chromic sulphate solution apparently contains not only a basic complex, but also a chromosulphuric acid, which is not precipitated by baric salts. This compound is only slightly occluded by baric sulphate because it is much dissociated. Its effect is simply to delay the precipitation of baric sulphate, a consequence which Küster and Thiel have correctly interpreted. The whole subject is now being further studied here, for the hypotheses are capable of further experimental treatment.

Harvard University, Cambridge, Mass.,
February 2, 1900.

THE ACTION OF AMMONIUM CHLORIDE UPON ANALCITE AND LEUCITE.

By F. W. CLARKE and GEORGE STEIGER.

In a recent paper (*Amer. Journ. Sci.*, October, 1899), upon the constitution of certain silicates, we showed that analcite, when heated with ammonium chloride, gave up a part of its soda, and absorbed ammonia to an appreciable extent. This result was so remarkable that it seemed to demand further investigation, upon material of different origin, and with variation in the details. The new experiments, which have led to highly interesting consequences, are now to be described.

The analcite previously studied was from Nova Scotia. To the kindness of President Regis Chauvenet, of the State School of Mines, we are indebted for a liberal supply of well-crystallised material from North Table Mountain, near Golden, Colorado, of which a uniform sample of about 80 grms. was prepared. Upon this sample the present series of experiments has been conducted. Analysis of the analcite gave the following results:—

Analysis.		Water by fractions.	
SiO_2	55·72	At 100°..	0·13
Al_2O_3..	23·06	„ 180°..	0·75
CaO	0·17	„ 260°..	2 44
Na_2O	12·46	„ 300°..	1 28
H_2O at 100° ..	0·13	„ 350°..	1 76
H_2O above 100°	8·26	„ redness	2·03
	99·80	Total ..	8·39

Above a low red heat no further loss of weight was observed. Upon boiling the powdered mineral for fifteen minutes with a 25 per cent solution of sodium carbonate, 0·45 per cent of silica was dissolved. After ignition, 0·57 per cent was soluble, which is practically the same amount. No silica was split off by heating.

The experiments with ammonium chloride fall into two series. The first of these was conducted precisely as in our former investigation, by grinding the powdered mineral into an intimate mixture with four times its weight of chloride, and heating in an open crucible. In three cases the material, after volatilisation of the ammonium chloride, was re ground with a fresh amount of the salt, and then heated again. The temperature and duration of the experiments was purposely somewhat varied. After heating, the material was leached out with water, the sodium chloride which had been formed was estimated, and in the residue the fixed ammonia was determined.

In this series there were four experiments, with results as follows:—

	Hours heated.	Temperature.	Soda removed.	Ammonia in residue.
A	28	300°	4·75	2·04
B	8½	350°	6·36	2·88
C	26	350°	3·76	1·72
D	5	340° to 380°	6·70	2·85

In our work upon the analcite from Nova Scotia the ammonia retained by the leached residue ranged from 2·03 to 2·36 per cent, while the extracted soda varied from 5·07 to 6·10. In two of the new experiments these figures are perceptibly exceeded, and they represent the shortest duration of heating. Prolonged heating seems to be undesirable, and to undo a part of the reaction which has taken place; otherwise the results obtained are of the same order as their predecessors. *About* one-half of the soda in the analcite is converted into chloride, while variable ammonia is retained.

In the second series of experiments a sealed tube was substituted for the open crucible. The powdered analcite was intimately ground with four times its weight of ammonium chloride, as before, and then heated to 350° in a tube furnace for from four to eleven hours. Under these conditions practically the whole of the soda in the mineral was converted into sodium chloride, while all of the liberated ammonia was absorbed by the residual silicate. Upon leaching the contents of the tube with water, to remove sodium and ammonium chlorides, a residue was obtained which exhibited constant composition whether dried at 100° or at the ordinary temperature of the air. Three samples of the residue were prepared and analysed; other samples were partially examined and used for subsidiary experiments. The three analyses, lettered for future reference, were as follows, the analcite itself being included in the table for comparison.

	Analcite.	Residue A.	Residue B.	Residue C.
SiO_2 ..	55·72	61·93	61·68	61·79
Al_2O_3 ..	23·06	25·21	25·33	25·24
CaO ..	0·17	—	—	—
Na_2O ..	12·46	0·40	0·22	0·28
NH_3 ..	—	7·23	6·95	7·71
H_2O ..	8·39	4·50	4·91	5·01
	99·80	99·27	99·09	100·03

Residue C was prepared with the greatest care, and was air-dried. Exposed over sulphuric acid in a vacuum desiccator for fourteen days, it lost in weight only 0·08 per cent. Tested for chlorine, only a slight trace could be recognised, but upon boiling for fifteen minutes with sodium carbonate solution it yielded 1·97 of soluble silica. After ignition only 1·70 of silica was soluble, or somewhat less than before. Upon heating at constant weight at 300°, only 0·46 per cent was lost; but at 350° it slowly decomposed, giving off ammonia. At 300° the compound is stable.

The 0·28 per cent of soda remaining in residue C may be regarded as representing unaltered analcite,—doubtless coarser particles which escaped complete transformation. It corresponds to 1·98 per cent of analcite, which, together with the 1·97 of soluble silica, and the 6·46 of water lost below 300°, may be deducted from the substance in order to obtain the composition of the definite compound. The latter amounts to 94·72 per cent of the total residue, and agrees very nearly in composition with the formula $2NH_3.H_2O.Al_2O_3.4SiO_2$. Re-calculating the 94·72 of residue to 100 per cent, we get the following comparison between analysis and theory:—

	Found.	Calculated.
SiO_2	61·07	60·92
Al_2O_3	26·15	25 88
NH_3	8·14	8·63
H_2O	4·64	4·57
	100·00	100·00

Written in rational form the compound becomes equivalent to an anhydrous ammonium analcite, $NH_4AlSi_2O_6$; that is, analcite in which sodium has been replaced by ammonium. From this point of view the reaction between analcite and ammonium chloride becomes a simple case of double decomposition, and is perfectly intelligible. To establish this conclusion, however, corroborative experiments were necessary.

In the first place, the observed equivalency between the sodium lost and the ammonia gained might be due to a mere coincidence, and so far be illusory. One atom of sodium, taking chlorine from ammonium chloride, liberates one molecule of ammonia, the amount which the analcite residue has retained. Suppose more ammonia were present,—could it be absorbed?

To answer this question another tube was prepared, with the usual mixture of analcite and ammonium chloride. This was covered by a loose plug of glass-wool, in front of which we placed enough pure lime to liberate about double the normal amount of ammonia. The tube was then sealed, and heated to 350° as in the previous experiments. Upon opening the tube, a strong outrush of ammonia was noticed; but in the leached and thoroughly washed residue only 7·52 per cent of ammonia was found. This quantity agrees with that from the previous samples, and shows that the limit of the reaction has been practically reached. One molecule of ammonia is retained, and no more.

Still another experiment was tried upon a portion of the residue marked C. If the compound is really an ammonium salt, it should be decomposable by caustic soda in such a way as to reverse the reaction by which it had been obtained. The substance, however, is very insoluble, so that the reaction takes place slowly. To phenol-phthalein it is absolutely neutral, and with Nessler's reagent it reacts only after long standing.

To settle the question a weighed portion of the compound was boiled in a distilling flask with a 10 per cent solution of sodium hydroxide, to which water was added from time to time. The distillate was collected in a tube containing aqueous hydrochloric acid, and the ammonia which passed over was weighed, ultimately as chloroplatinate. By four hours boiling 6·90 per cent of ammonia was driven off and determined; and the residue remaining in the flask, after washing until no alkaline reaction could be detected in the wash-water, was examined for soda, of which 10·41 per cent was found. The anticipated reaction had taken place, although not completely; it was enough, however, to confirm our opinion, and to establish the nature of the new compound beyond reasonable doubt. Other confirmation was obtained later, from the study of leucite.

The foregoing paragraphs now enable us to understand a phenomenon which we observed in our work with the open crucible. In that case a partial reaction takes place between the analcite and the ammonium chloride, pro-

ducing, as in the sealed tube, a mixture of an ammonium alumino-silicate with sodium chloride, the two substances being separable by leaching. But if, instead of leaching, the mixture be heated to full redness, ammonium chloride is re-formed and given off, leaving a residue which contains little or no sodium chloride, and is wholly insoluble, or almost so, in water. That is, the reaction which occurs at 350° is reversed at the higher temperature, and anhydrous analcite, or an isomer of it, is regenerated. Ammonium and sodium again change places, and the original state of molecular equilibrium is restored.

What, now, is the nature of the product obtained in the open crucible, after sodium chloride has been removed? It is a definite, intermediate compound, or an indeterminate mixture? In our former paper we adopted the first of these alternatives, and assigned to the substance the formula $H_2Na_2Al_4Si_8O_{24}.NH_3$. In this expression we were influenced by the researches of Friedel, who had shown that ammonia could in part replace the "zeolitic" water of analcite; but it now appears that the phenomenon observed by him is quite distinct from that discovered by us, and, indeed, of an entirely different order. We may therefore, in accordance with our new data, rearrange the formula; transforming it to that of an ammonium salt, $HNa_2NH_4Al_4Si_8O_{24}$, the agreement with the analytical figures being approximate only. The results obtained are not sharp enough for certainty.

This product we are now inclined to regard as a mixture, although it is not strictly intermediate between analcite and its final ammonium derivative. Only half of the eliminated sodium has been replaced by ammonium, while hydrogen, or water, makes up the deficiency. It seems probable that the reaction in the sealed tube and that in the open crucible are at first essentially the same; but that in the latter case secondary reactions follow which cause the variations in the final results. In the sealed tube, the element of pressure comes into play, and the reaction is complete. In the open crucible, pressure is lacking; some ammonia escapes fixation and reacts upon a part of the sodium chloride which was at first formed; hence the composition of the leached residue is essentially modified. This residue may be a definite compound; but the case in its favour is unproved, and the presumption is rather against it.

Between analcite and leucite the closest analogies have long been recognised. The two minerals have similar composition, they resemble each other in crystalline form, and they yield, upon alteration, products of the same order. Recently also, analcite, like leucite, has been identified as a not uncommon constituent of volcanic rocks; analcite-basalt being a good example. In view of these resemblances, it was plainly desirable to compare the minerals by means of the ammonium chloride reaction; a task which has been performed with satisfactory results.

In a preliminary experiment a sample of leucite, taken without regard to purity, was heated with ammonium chloride to 350° in a sealed tube. Potassium chloride was formed corresponding to 18·06 per cent of potash, and in the leached residue 6·90 per cent of ammonia was found. The foreseen reaction had occurred, and more careful work was accordingly undertaken.

Our material consisted of a large irregular crystal of leucite from Vesuvius, which yielded about 20 grms. of the pure mineral. This was ground to a uniform sample, and a portion of it was analysed; the analysis will be given presently. The sealed tube experiments were conducted precisely as in the case of analcite, and they confirmed both the preliminary test and our anticipations. Chlorides were formed equivalent to 18·53 per cent of potash, 1·08 of soda, and 0·08 of alumina; the reaction therefore was very nearly complete. The leached residue was then analysed, and the data, compared with the analysis of the original mineral, were as follows. (See Table, next column).

	Leucite.	Residue.
SiO_2	55·40	60·63
Al_2O_3	23·69	26·44
CaO	0·16	trace
K_2O	19 54	0·50
Na_2O	1·25	0·25
NH_3	—	7·35
H_2O	0·24	5·17
	100·28	100·34

Leucite, then, gives the same reaction as analcite, and yields the same ammonium compound. A closer agreement in the composition of the latter could not reasonably be demanded. Ammonium leucite is formed in both cases, by ordinary double decomposition, in a state of approximate purity; the first silicate of ammonium, we think, which has ever been prepared. At the suggestion of Dr. F. K. Cameron an attempt was made to transform ammonium leucite into the corresponding lime salt, $CaAl_2Si_4O_{12}$, by fusion with calcium chloride. The ammonium leucite was mixed with a saturated solution of calcium chloride, which was evaporated to dryness, then heated gradually to dehydration, and finally fused. Ammonium chloride was given off and identified. Upon treating the fused mass with water, filtering, and thoroughly washing the residue, a white powder was obtained, which, after drying at 100°, was analysed. It was also examined microscopically by Mr. J. S. Diller, who found it to consist of apparently isotropic grains, showing traces of incipient crystallisation. The following analysis is contrasted with the theoretical composition of calcium leucite, from which it varies considerably:—

	Found.	Calculated.
SiO_2	54·35	60·30
Al_2O_3	26·23	25·63
CaO	17·38	14·07
K_2O	0·16	—
Na_2O	0·25	—
Cl	0·28	—
Loss on ignition	1·24	—
	99·89	100·00

Evidently, the desired salt was not definitely obtained, and the product appears to be a mixture. The reaction, however, tends in the right direction, and deserves further study under other conditions. Probably the water which was present in the mixture of silicate and chloride took part in the changes produced; although of this we cannot be certain. It is interesting to note that the product obtained approximates in composition to the meteoritic mineral maskelynite, which is regarded as perhaps a calcium leucite by Groth.

The question now arises whether the observed reaction with ammonium chloride is limited to a few species or is fairly general. To test this point we have made preliminary experiments upon a number of other minerals, heating in a sealed tube to 350°, with the following results:—The percentage of bases removed is calculated in each case upon the original minerals; that of the ammonia is the amount found in the leached and washed residue.

Natrolite.—17·56 per cent of soda removed, 8·29 of ammonia taken up.

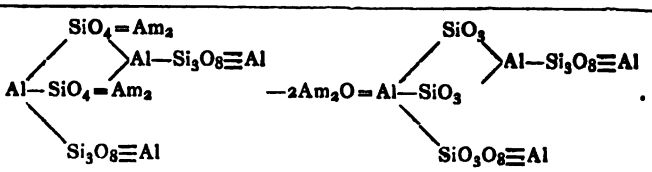
Laumontite.—4·51 of lime and 0·35 of soda removed, 3·95 of ammonia absorbed.

Stilbite.—6 54 of lime and 1·31 of soda extracted, 5·36 of ammonia in the residue.

Chabasite.—4·37 of lime and 1·13 of soda removed, 4·55 of ammonia retained.

Thomsonite.—7·94 of lime and 3·40 of soda extracted, 4·01 of ammonia taken up.

Heulandite.—4·59 of lime and 1·59 of soda extracted, 3·64 of ammonia retained.



Apophyllite.—21·59 of lime and 5·18 of potash removed, 0·79 of ammonia in residue.

Pectolite.—20·72 of lime and 6·46 of soda extracted, 1·44 of ammonia retained.

Elæolite.—2·25 of soda taken out, 0·74 of ammonia retained.

It will at once be seen that the reaction takes place in most cases, but with varying completeness. In some instances the decomposition is very slight, in others it is near the possible limit. In each case the ammonia of the residue was obtained by decomposing the material with hydrochloric acid, supersaturating with caustic soda, distilling into tubes containing hydrochloric acid, and final weighing as ammonium chloroplatinate. Each of the minerals mentioned is to be studied more thoroughly hereafter, and prehnite, sodalite, and possibly other silicates will be added to the list.

It is evident that all of the zeolites are well suited to this new method of study, and it is more than probable that the differences in the action of ammonium chloride upon them correspond to differences in their chemical structure. But the reaction goes further than this, for it enables us to substitute in many minerals a volatile base for fixed bases, yielding silicates which split up on ignition in such a way as to shed light upon their molecular constitution.

For example, if ammonium leucite is a true metasilicate, a salt of the acid H_2SiO_3, it should break up, when ignited, in accordance with the following equation:—

$$2NH_4Al(SiO_3)_2 = Al_2(SiO_3)_3 + 2NH_3 + H_2O + SiO_2;$$

that is, one-fourth of a silica ought to be set free, measurable by extraction with sodium carbonate solution. No such splitting off occurs, however: an ammonium leucite which already contained 1·97 per cent of soluble silica gave only 1·70 per cent after ignition; hence no additional silica had been liberated. We may conclude therefore that analcite and leucite are not true metasilicates but pseudo compounds; either salts of a polymeric metasilicic acid or mixtures of ortho- and tri-silicates as suggested in our former paper. The ammonium salt corresponding to such a mixture, when ignited, might be expected to give the accompanying reaction (see above)—a reaction which is in harmony with our experimental results. In it no free silica appears; and many, if not all, conditions of the problem are satisfied. Other formulæ, however, are possible; so that these offer merely one explanation of the phenomena, which one may or may not be the best.

Action of Low Temperatures on some Cyclic Compounds.—V. Markovnikof.—The author has tried the effect of low temperatures for purifying the naphthenes extracted from Russian petroleums and the derivatives of these bodies, by means of crystallisation. Hexanaphthene (boiling at 80–82°) crystallises at about −46°; after three crystallisations it solidifies at −11°, as a white mass apparently formed of leaf-like crystals very different to those of benzene. The ketone, $C_6H_{10}O$, crystallises at −45°; the corresponding alcohol at the ordinary temperature; nitrohexanaphthene at −34°. Heptanaphthene, metaoctonaphthene, pentamethylene, and methylpentamethylene do not solidify at −79°, but certain of their derivatives crystallise in solid CO_2. This method enables us to detect traces of impurities which have escaped notice during analysis.—*Journ. Soc. Phys. Chim. R.*, xxxi., p. 356.

NOTICES OF BOOKS.

Optical Activity and Chemical Composition. By Dr. H. LANDOLT. Translated with the Author's permission by JOHN MCCRAE, Ph.D. London and New York: Whittaker and Co. 1899. Pp. 158.

THE contents of this volume form the eighth chapter of the first volume of Graham-Otto's "Lehrbuch der Chemie," with notes and additions thereto by the translator.

Optical activity is the power possessed by a number of bodies of being able to rotate the plane of polarisation of a plane-polarised ray of light, which is passed through them; there are two types of optical activity called *crystal* rotation, and *liquid* or *molecular* rotation.

In 1848 Pasteur made his well known discovery of the right and left-handed rotation of the crystallised sodium ammonium salt prepared from racemic acid. The crystals which cause this double rotation are said to be *enantiomorphic*. By these and other observations Pasteur introduced into science a new conception, that of molecular asymmetry; the van 't Hoff and Le Bel theories, which have grown out of Pasteur's original ideas, have been brilliantly confirmed by later experience. The second part of the book deals with this subject in a very clear and succinct manner, showing the connection between the rotatory power and the chemical composition of carbon compounds, while the third part deals with the degree of rotation and chemical constitution. There is on page xi. an addendum to pp. 121—131, which should not be overlooked.

An Introduction to Analytical Chemistry. By G. G. HENDERSON, D.Sc., M.A., F.I.C., and M. A. PARKER, B.Sc. London, Glasgow, and Dublin: Blackie and Son, Limited. 1899. Pp. 228.

THIS introduction to analytical chemistry will be found useful by students working under a demonstrator, who will first put them through a series of simple chemical experiments illustrative of chemical theory, such as the preparation of gases, the determination of densities, &c., before beginning the course of regular quantitative analysis; some such general knowledge of the subject being essential to a proper comprehension of group analysis, which is then fully described and illustrated.

Exercises in Practical Physics for Schools of Science. By R. A. GREGORY and A. T. SIMMONS, B.Sc. (Lond.). In two parts. London: Macmillan and Co., Limited. 1899. Pp. 374.

THE advantages claimed by the authors of this work are the number and variety of the exercises used to exemplify each of the principles dealt with; the limitation of the text to instructions necessary for the performance of the experiments and for understanding the meaning of the results; the number of new and simple experimental devices it has been found possible to make use of, and the numerous illustrations which show at a glance the apparatus required and the method of procedure. The authors by no means claim too much, as we find the exercises and experiments are very well chosen, the descriptive portions of the book is clear and precise, while the illustrations though numerous are good, and will

materially help the student to follow the instructions given.

Lexicon of the Carbon Compounds. (Lexikon der Kohlenstoff-Verbindungen). By M. Richter. In thirty-nine parts. Hamburg and Leipzic: Leopold Voss. London and Edinburgh: Williams and Norgate. 1899. Parts 13 to 39. Pp. 2482.

The first twelve parts of this excellent work have already been noticed in our columns (vol. lxxx., p. 229), the remaining parts, making thirty-nine in all, have now been published, and show that the author's forecast of about 65,000 carbon compounds, has been considerably exceeded, no fewer than 74,174 being now registered, so continuous and increasing are the fresh discoveries constantly being made in this far-reaching subject.

The two volumes now completed form a monument of patience and industry, and must be of immense value to scientific chemical research.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 13, March 26, 1900.

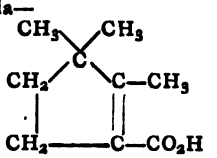
Deviation of Radium Rays in a Magnetic Field.—Henri Becquerel.—M. and Mdme. Curie have recently shown that the rays emitted by radium carry negative electric charges. The author continues the research by measuring the electrostatic deviation of the rays. This measurement is particularly interesting because it allows of the determination of the velocity of translation of the electric charges and the proportions of the material masses involved in the charges transmitted. The arrangement consists essentially in allowing a very narrow pencil of rays to pass between two little charged plates, and receiving the pencil on a photographic plate enclosed in black paper. The fine metallic wires placed in front of the sensitive plate give shadow lines, which allow of accurate measurement. It is found that the pencil emitted by radium is repelled by the negatively charged plate. By reversing the direction of the field, the displacements are reversed. The author used for his experiments two small rectangular copper plates 3·45 c.m. high. These were placed vertically and fixed, by means of paraffin, so as to leave a space, 1 c.m. thick, of air between them. In this space the electric field was measured by the difference of potential between the plates in C.G.S. units. One of the plates was to earth and the other to a battery of six large jars, whose capacity considerably reduced the velocity of the variation of potential between the two plates under the influence of radium. A Henley's electrometer in connection with the battery had been previously graduated by observation, so as to give approximately the potential of the battery. During the experiment this potential was kept constant by constantly re-charging the battery by an influence machine. The photographic plate, wrapped in black paper, was fixed outside the field, the radio-active material being placed below the field, in a little lead vessel. By this arrangement, numbers were obtained from which the author calculates the deviation of the rays.

Selenide of Zinc and its Dimorphism.—M. Fonzes-Diacon.—The author prepares crystallised zinc selenide (*a*) by the action of seleniuretted hydrogen on zinc chloride; (*b*) by the reduction of zinc selenate under the influence of carbon and hydrogen; and (*c*) by the fusion of precipitated zinc selenide in the electric furnace. He obtains hexagonal zinc selenide constituting the dimorphous form of cubic selenide prepared by M. Margottet. By a similar method cadmium selenide can be prepared in the hexagonal system.

Hydrated Peroxides of Barium.—M. de Forcrand.—In a former communication on the hydrated barium peroxides, the author based all his calculations on a manifestly inexact assumption, owing to an error of sign: $BaO.9H_2O$ + water = + 14·1 cal. He therefore repeats the experiments and calculations, and gives the corrected numbers.

New Chlor-compound of Mercury and Antipyrine.—J. Ville and Ch. Astre.—Hirsch and C. Schuyten have announced the preparation and properties of halogen mercuric derivatives of antipyrine corresponding to the general formula $C_{11}H_{12}N_2O.HgR_2$. The corresponding iodine compound is not known, in spite of many attempts to prepare it. Whilst making another attempt to realise this iodine compound, the author was led to obtain some new halogen derivatives of antipyrine corresponding to the general formula $2(C_{11}H_{12}N_2O).HgR_2.HR$; in which R represents the monovalent halogen radicle. The chlor-compound was obtained by the action of mercuric chloride on antipyrine in presence of sodium chloride and hydrochloric acid; this latter being indispensable for obtaining a crystalline product.

Constitution of Isolauronic Acid.—G. Blanc.—In a recent paper, the author confirmed his statement that the constitution of isolauronolic acid ought to be represented by the formula—



contrary to the theories of W. H. Perkin, jun. The present paper establishes the above formula in a definite manner. When isolauronic acid is treated with sodium amalgam in alkaline solution, dehydro-isolauronic acid is obtained, $C_9H_{14}O_3$. This acid is a γ-ketonic acid, and not a δ-acid alcohol, as Perkin affirmed.

MISCELLANEOUS.

The Chemical Society of Paris.—It is officially announced that an international Banquet will be held by the Chemical Society of Paris in honour of those gentlemen who, by their presence at the Universal Exhibition, will represent pure and applied chemistry. The date fixed for the banquet is Thursday the 19th July, when the chair will be taken by M. Berthelot, Honorary President.

Royal Institution.—On Tuesday next, April 24th, Dr. H. R. Hill will deliver the first of a course of three lectures at the Royal Institution on "Studies in British Geography." On Thursday, April 26th, Professor Dewar will commence a course of four lectures on "A Century of Chemistry in the Royal Institution." On Saturday, April 28th, Professor Stanley Lane-Poole will deliver the first of two lectures on "Egypt in the Middle Ages." The Friday Evening Discourse on April 27th will be delivered by Lord Kelvin, the subject is "Nineteenth Century Clouds over the Dynamical Theory of Heat and Light." The discourse on May 4th is on "Pottery and Plumbism," and the lecturer will be Professor T. E. Thorpe.

The Nitrification of Organic Nitrogen.—V. Omelianski.—According to the experiments made by the author, pure cultures of the nitrifying bacteria have no action on the nitrogen of organic compounds. Contrary to what has been alleged, this negative result is not due to too great a concentration. The oxidation of the organic nitrogen requires that this element should be first transformed into ammonia by the co-operation of an organism

capable at least of effecting the ammoniacal fermentation of the organic compounds. The opposite conclusions formulated by Frankland, Warington, and Stutzer are, according to the author, based on errors of observation. The author confirms his negative conclusions by a very complete series of synthetic experiments. He took pure cultures of *B. racemosus*, of *B. nitrosomonas*, and of *B. nitrobacter*, and cultivated them together in ordinary alkaline broth. With *B. racemosus*+*nitrosomonas*+*nitrobacter*, the nitrification is accomplished right up to nitric acid, passing through ammonia and nitrous acid. With *B. racemosus*+*nitrosomonas*, the action stops at nitrous acid. With *B. racemosus*+*nitrobacter* only ammonia is formed, since the absence of organisms giving rise to nitrous acid suppresses the alimentation of the nitrifying organism. Finally, with *B. nitrosomonas*+*nitrobacter* no action whatever was observed, even after the lapse of six months.—*Centralbl. f. Bakter.*, 1899, p. 473.

Transformation of Trimethylene into Propylene.—S. Tanatar.—The author has observed that by very rapidly passing a mixture of trimethylene and propylene into cold bromine, the second is totally absorbed, while the first is recovered almost integrally; in the same way we can, by submitting trimethylene to the successive and repeated action of bromine with heat, completely transform it into bromide of propylene. Trimethylene being soluble in sulphuric acid, the author prefers the bromine method for demonstrating the transformation of trimethylene into propylene under the influence of heat. He uses a single bromine wash bulb, placed in the dark, followed by a soda wash-bulb; the speed of the current was 6 litres per hour. Under these conditions, after four hours, while operating on 7·5 litres of purified trimethylene with bromine, and avoiding all loss due to solubility, almost the theoretical quantity of bromide of propylene boiling at 141—143°, was obtained. A comparative test, made with pure trimethylene, showed that this latter is not absorbed by bromine to an appreciable extent except at 20° in sunlight; the products of the reaction consist essentially of bromide of trimethylene boiling at 155—156°, and small quantities of the products above and below.—*Berichte*, xxxii., p. 1965.

Combinations of some Organic Acids with Sulphuric Acid.—S. Hodgewerff and W. A. van Dorp.—Certain acids dissolve in sulphuric acid (at 96 per cent H_2SO_4), giving compounds of addition; on being cooled in ice the compounds crystallise. We thus obtain—

$(CH_2—CO_2H)_2.SO_4H_2$;
$(=CH—CO_2H)_2=2SO_4H_2$;
$[CO_2H—CH=C(CH_3)—CO_2H]SO_4H_2$ (mesaconic);
$C_6H_5—CO_2H.SO_4H_2$;
$2C_6H_5—CH=CH—CO_2H.3SO_4H_2$; and
$C_6H_5—CH=CH—CO_2H—SO_4H_2$.

These bodies are very unstable, and are decomposed by all liquids.—*Revue Trav. Ch. P.-B.*, xviii., p. 211.

Obstacle opposed to Chemical Reactions by the Positions of Certain Groups.—C. Paal and F. Härtel.—Following up their previous research, the authors have now been occupied with the examination of the *o* oxybenzylised bases; one of the authors, with M. Benker, has already shown that *p*-nitrobenzyl-*o*-nitroaniline, as well as *o*-nitrobenzyl-*o*-nitraniline, were not able to furnish acetylised derivatives, while the corresponding *m*- and *p*-derivatives gave them easily. It was found advisable to repeat the experiments with the *o*-oxybenzylised bases, and with this end in view the authors have prepared the three *o*-oxybenzylnitranilines. Experiments made with these bases have shown that the *o*-oxybenzyl-*m*- and *p*-nitranilines may be acetylised either in the hydroxyl group or in the iminic residue, while in *o*-oxybenzyl-*o* nitraniline the acetyl group only enters the hydroxyl. With regard to the colour of the compounds, it has been remarked that *o*-oxybenzyl-*o*-nitraniline, which is red, gives a yellow acetyl derivative, while the mono- and the di-substituted products of *m*- and *p*-nitranilides are colourless. The introduction of the acetyl residue into the phenolic hydroxyl apart from the chromophore "nitro" group only weakens the shade, while the substitution in the iminic residue, joined to the same nucleus as the nitro group, completely neutralises it. The bases examined have been prepared by the condensation of *o*-oxybenzylic alcohol (saligenine) with the three nitranilines; they behave generally, from the point of view of acetylation, in the same way as the nitrobenzylised nitranilines. Full details of their preparation and properties are given in the paper.—*Berichte*, xxxii., p. 2057.

MEETINGS FOR THE WEEK.

TUESDAY, 24th.—Royal Institution, 3. "Studies in British Geography," by Hugh Robert Mill, D.Sc., LL.D., &c.

WEDNESDAY, 25th.—Institution of Mining and Metallurgy, 8. "The Electrical Precipitation of Gold on Amalgamated Copper Plates," by Dr. T. Kirke Rose. "Notes on the Namaqualand Copper District," by J. A. Chalmers. A Note on Hand Concentration Tests," by Walter McDermott "Notes on the Elmore Concentrating Process," by C. M. Rolker.

THURSDAY, 26th.—Royal Institution, 3. "A Century of Chemistry in the Royal Institution," by Prof. Dewar, M.A., LL.D., F.R.S.

FRIDAY, 27th.—Royal Institution, 9. "Nineteenth Century Clouds over the Dynamical Theory of Heat and Light," by Lord Kelvin, G.C.V.O., D.C.L., LL.D., F.R.S.

— Physical, 8. (In the Solar Physics Observatory, Exhibition Road, South Kensington). "A Short Account of the Physical Problems now being Investigated at the Solar Physics Observatory, and their Astronomical Applications," by Sir Norman Lockyer. Weather permitting, the 36-inch, 10-inch, and 9-inch telescopes will be used for the observation and photography of celestial objects and their spectra. The Apps-Spottiswood coil and 21-ft. Rowland grating will also be in operation.

SATURDAY, 28th.—Royal Institution, 3. "Egypt in the Middle Ages," by Prof. Stanley Lane-Poole, M.A.

ROYAL INSTITUTION OF GREAT BRITAIN,
ALBEMARLE STREET, PICCADILLY, W.

Professor DEWAR, M.A., LL.D., F.R.S., M.R.I., Fullerian Professor of Chemistry, R.I., will, on THURSDAY next, APRIL 26th, at THREE o'clock, begin a course of Four Lectures on "A CENTURY OF CHEMISTRY IN THE ROYAL INSTITUTION."

Subscription to the Course, Half a Guinea. To all the Courses in the Season, Two Guineas.

Tickets may be had at the Office of the Institution.

THE LONDON HOSPITAL MEDICAL COLLEGE.

The SUMMER SESSION COMMENCES on MAY 1.

The Hospital is the largest in the Kingdom; nearly 800 beds are in constant use, and no beds are closed. Being the only general hospital for East London—*i.e.*, for a million and a half people—the practice is immense. In-patients last year, 11,622; out-patients, 178,838; accidents, 17,370; major operations, 2320.

APPOINTMENTS.—Owing to the enormous number of patients, more appointments are open to Students than at any other hospital. Sixty qualified appointments are made annually, and more than 150 Dressers, Clinical Clerks, &c., appointed every three months. All are free to Students of the College. Holders of resident appointments have free board.

SCHOLARSHIPS AND PRIZES.—Twenty-seven Scholarships and Prizes are given annually. Students entering in May can compete for five Entrance Scholarships in September.

Special arrangements have been made to enable Students entering in May to present themselves for examination in Chemistry, &c., in July.

SPECIAL CLASSES are held for the University of London and other higher Examinations. Special entries for Medical and Surgical Practice can be made. Qualified practioners will find excellent opportunities for studying the rarest diseases.

A reduction of 15 guineas is made to the sons of members of the profession.

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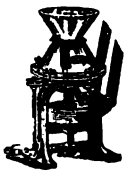

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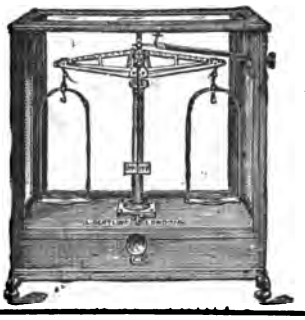

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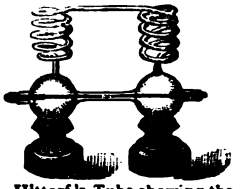

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ON THE WEIGHT OF HYDROGEN DESICCATED BY LIQUID AIR.*

By Lord RAYLEIGH, F.R.S.

In recent experiments by myself and by others upon the density of hydrogen, the gas has always been dried by means of phosphoric anhydride; and a doubt may remain whether, on the one hand, the removal of aqueous vapour is sufficiently complete, and, on the other, whether some new impurity may not be introduced. I thought that it would be interesting to weigh hydrogen dried in an entirely different manner, and this I have recently been able to effect with the aid of liquid air, acting as a cooling agent, supplied by the kindness of Professor Dewar from the Royal Institution. The operations of filling and weighing were carried out in the country as hitherto. I ought, perhaps, to explain that the object was not so much to make a new determination of the highest possible accuracy, as to test whether any serious error could be involved in the use of phosphoric anhydride, such as might explain the departure of the ratio of densities of oxygen and hydrogen from that of 16 : 1. I may say at once that the result was negative.

Each supply consisted of about 6 litres of the liquid, contained in two large vacuum-jacketted vessels of Professor Dewar's design, and it sufficed for two fillings with hydrogen at an interval of two days. The intermediate day was devoted to a weighing of the globe *empty*. There were four fillings in all, but one proved to be abortive owing to a discrepancy in the weights when the globe was empty, before and after the filling. The gas was exposed to the action of the liquid air during its passage in a slow stream of about half a litre per hour through a tube of thin glass.

I have said that the result was negative. In point of fact the actual weights found were 1/10 to 2/10 m.grms. *heavier* than in the case of hydrogen dried by phosphoric anhydride. But I doubt whether the small excess is of any significance. It seems improbable that it could have been due to residual vapour, and it is perhaps not outside the error of experiment, considering that the apparatus was not in the best condition.

NOTE ON THE DETERMINATION OF MANGANESE AS SULPHIDE.

By J. and H. S. PATTINSON.

In determining the amount of manganese in manganese ores, &c., by the sulphide method care should always be taken to treat the filtrates from the sulphide of manganese precipitates so as to obtain the manganese which they may still hold in solution. We find that when all the precautions to secure the complete precipitation of the manganese from its solutions by sulphide of ammonium described in Fresenius's "Quantitative Analysis" have been carefully observed, the filtrates still invariably contain some manganese in solution, and sometimes a notable quantity. This can be ascertained and the amount determined by acidifying the filtrate, oxidising precipitated sulphur by bromine, and then adding excess of ammonia and bromine to precipitate the manganese as oxide. The manganese thus obtained should, of course, be added to the principal sulphide precipitate.

The final precipitate of sulphide of manganese should also always be examined as to its purity, for we find that it very often contains small quantities of barium, cobalt, iron, silica, &c., sufficient to materially affect the result even although the solution of the manganese ore has been specially treated to eliminate these constituents before the sulphide of ammonium is added. In ores containing much lime or magnesia the sulphide precipitate may also contain these substances.

If the precautions above mentioned are not attended to, the correct amount of manganese contained in an ore will not be obtained by the analyst, unless, by chance, the errors of deficiency and excess should exactly neutralise each other.

ON THE SEPARATION OF NICKEL AND IRON WITH AMMONIA

By FRED IBBOTSON and HARRY BREARLEY.

A good illustration of the difference of opinion which may be held of a simple analytical process is afforded by the separation of iron and nickel by means of ammonia. To "precipitate with ammonia, filter off the iron, &c.," appears to be such a matter as one could not get wrong about. Amongst the published and private opinions we have gathered, however, are all kinds ranging from "absolutely worthless" to "thoroughly trustworthy." In operating under varying conditions we have realised these extreme opinions, and arrived at a fairly simple explanation of the differences.

By pouring an excess of ammonia into an aqua regia solution of 1 grm. of a 10 per cent FeNi alloy, or by pouring the alloy solution into the ammonia, the Ni in the filtrate would vary from 3 to 30 per cent of the correct amount, according to the excess of ammonia used; these results justify the expression "absolutely worthless."

A number of imperfect separations are popularly explained by supposing that at the moment a precipitate is formed it exerts some peculiar influence on the element which should remain in solution, and partly "carries it down;" but in this case, if the iron seized on the nickel at the moment it becomes precipitated as $Fe_2(HO)_6$, we might properly expect to find that after careful neutralisation, *i.e.*, formation of $xFe_2Cl_6yFe_2(HO)_6$, the nickel carried down would be less, because the ferric chloride remaining to be decomposed is less. There is no foundation for such theorising; such a modification does not at all improve the separation.

If we precipitate a solution of ferric chloride with an excess of ammonia, add a faintly acid or ammoniacal solution of nickel chloride, shake and filter, we find almost as little nickel in the filtrate as though the nickel had been present when the iron was precipitated; this shows that the act of *forming* $Fe_2(HO)_6$ has nothing to do with the imperfect separation. The error arises through the absorption of the ammoniacal nickel solution by the ferric hydrate, just as sand, filter-paper, &c., will extract particular salts from their solutions, or from mixtures of salt solutions.

Whatever modifies the precipitated ferric hydrate or the ammoniacal nickel solution may modify the separation. To this end we have precipitated with ammonia in the presence of such acids as would form basic salts with the iron, such as phosphates, chromates, molybdates, &c.; the separations were mostly worse with than without this modification.

The most promising modifications have to do with the ammoniacal nickel solution; this is recognised, with more or less reason, by the practice of adding salts of ammonia before precipitating the iron. The value of adding such salts was illustrated by neutralising 50 c.c. ammonia

* Read before the Royal Society, April 5, 1900.

(0·880) to $(NH_4)Cl$, $(NH_4)NO_3$, and $NH_4)_2SO_4$ respectively, adding a solution containing 1 grm. of iron and 0·1 grm. of nickel, and precipitating at boiling point with the same amount of ammonia in each case; the amounts of nickel remaining in solution are shown in the table:—

Ammonia salt.	Grm. Ni recovered.
Chloride	0·0607
Nitrate	0·0584
Sulphate	0·0573
None	0·0083

By keeping all else constant the results are better as the ammonia salt is increased or as the excess of ammonia is increased, so that by using about 100 grms. of ammonium chloride and about 50 c.c. strong ammonia in excess we may obtain a "thoroughly trustworthy" separation. The following results were obtained by using large amounts of ammonium sulphate and ammonia, the amount of iron in each case being 1 grm.:—

Ni added.	Ni found.
0·01	0·0098
0·02	0·0197
0·03	0·0300
0·05	0·0497
1·00	0·9980

In order to effect an accurate separation by the use of ammonia salts, much larger amounts must be added than are needed to form the respective chloride, nitrate, or sulphate of nickel ammonia. Except economically there is no disadvantage in this so long as the subsequent estimation of the nickel is not interfered with; by substituting potassium cyanide, however, a perfect separation can be effected when very little more KCN is present than is needed to form $Ni(CN)_2.2KCN$. Thus, the decigrm. of nickel mixed with 1 grm. of iron required 66·4 c.c. KCN; with the varying amounts of KCN shown below the nickel which remained in solution when the mixture is poured into an excess of ammonia is as indicated.

C.c. KCN used.	Grm. Ni recovered.
70	0·09991
100	0·09985
150	0·09994

In the above series the nickel was estimated cyanometrically after boiling the acidified solution so as to decompose the double cyanide and drive out the HCN. The cyanide is not, we are informed, a convenient solution from which to precipitate nickel electrostatically, but such as are wedded to gravimetric processes will find that after precipitating the iron with caustic soda instead of ammonia the cyanides are readily decomposed, and the Ni_2O_3 precipitated with bromine (and soda if necessary).

If a standard solution of potassium cyanide be used in this last described process it is quite feasible to estimate the excess of KCN in an aliquot part of the filtrate, by means of silver nitrate and potassium iodide, and so calculate the amount of nickel present in the alloy. This is the most rapid process we know of estimating nickel in ferro-alloys which includes also a separation of the iron; we have used it with satisfaction for some years. With care to dissolve always in the same amount of acid the operations may be concluded in the same time as is taken to make an Eggertz carbon test on the stage of a Siemens furnace, and as it is not interfered with by the presence of chromium it is well suited to the circumstances under which such tests are required, *i.e.*, in the manufacture of ordnance. An outline of the process is: Add ammonia to the acid solution of the sample until at least all the free acid is neutralised, add an excess of standard KCN, pour into an excess of ammonia, and titrate a portion of the filtrate with $AgNO_3$ in the well-known manner. Such an excess of ammonia as makes the filtrate not too alkaline for the subsequent titration is ample; the excess of KCN should not be needlessly large. Some examples are:—

Per cent Ni present.	Per cent Ni found.
5·0	5·03
3·0	3·00
1·0	1·02
35·0	35·07
73·5	73·5

In nearly every case the steel-works analyst, to whom these rapid methods chiefly appeal, knows to 1 per cent or so how much nickel is present in the materials being examined, and the excess of KCN can be added accordingly, but where the percentage may be 5 or 50 the process is less suited because the excess of KCN adheres to, or combines to, a slight extent with the precipitated iron, and when the excesses are very large this error becomes appreciable. As an example, we quote a 3 per cent nickel steel which was purposely treated with varying excesses of KCN:—

Percentage excess of KCN.	Per cent Ni found.
5	3·00
25	3·03
50	3·04
150	3·15

Nickel is absorbed by precipitated ferric hydrate to an appreciable extent from ammoniacal solutions only; in fact, the nickel thus removed from solution is largely redissolved on making the solution so slightly acid that the $Fe_2(HO)_6$ is hardly at all dissolved, and separations have been based on this fact. With like reason, when the iron is precipitated in the presence of ammonium chloride with so little ammonia that finally the filtrate is distinctly acid, the separation is a good one. The Schwarzenberg separation is but a particular mode of performing this operation; it depends on the nice adjustment, in the cold solution, of the $xFe_2Cl_6.yFe_2(HO)_6$ compound, so that when the ammonia is no longer to be added, to use Fresenius's words, "the fluid fails to recover its clearness after standing some time in the cold, but, on the contrary, becomes rather more turbid than otherwise."

Perhaps the least troublesome way of making this separation is to heat the acid solution of iron and nickel to boiling-point. At this temperature the ferric hydrate first precipitated is easily dissolved by the undecomposed ferric chloride, and very little time is lost in obtaining a turbid solution, which, however, does not hold any real filtrable precipitate, and is really obtained with less ammonia or ammonium carbonate than would be needed to form a lasting precipitate in the cold solution. From this point, only a very dilute solution of ammonia should be used, and when the total ammonia added is equal to, or somewhat less in amount than would be needed to form a permanent turbidity if the neutralisation had been made in the cold solution, the iron will have been precipitated.

The operation requires considerable care and attention, but is not otherwise a difficult one; its success depends on keeping the solution acid throughout. If the solution be ever so slightly alkaline, the separation is not nearly perfect. Appended are examples of separations made as above, but omitting the addition of ammonium chloride (II.); in the same way, but adding 2 or 3 c.c. acetic acid after precipitating (I. and V.); as in II., but adding a few c.c. acetic acid before precipitating (III. and IV.). The process could be modified in various other ways without affecting the accuracy of the separation.

	Present.		Nickel found.
	Fe. Grms.	Ni. Grm.	Grm.
I.	1	0·20	0·1999
II.	1	0·10	0·0998
III.	1	0·05	0·0497
IV.	2	0·10	0·1002
V.	2	0·03	0·0298

Technical Department,
University College, Sheffield.

NEW METHOD FOR THE ESTIMATION OF PHOSPHORIC ACID IN PHOSPHATES.

By W. HOFFMEISTER.

The author has given up the use of citric acid as a solvent for phosphoric acid in bone ashes, and all phosphates in general, with the exception of Thomas's scoria. He proposes the use of an ammoniacal solution of humic acid in which the phosphate is suspended and carefully stirred, at the same time passing a current of carbonic acid through the liquid.

Into a 2 litre flask, in which a little sand has been placed, is poured a solution of 15 grms. of humic acid in dilute ammonia, 5 grms. of the phosphate under examination, and at least 1 litre of water. A current of carbonic acid is passed through the liquid for twelve hours, adding a little ammonia from time to time. The liquid is then poured into another flask, the sand is washed, and the wash-waters added to the original liquid; the whole is then made up to a known volume, and filtered. Four-fifths of the volume are then taken and evaporated to dryness on the water-bath with a little hydrochloric acid, the residue is taken up with water and hydrochloric acid, filtered, washed, and the clear solution then made up to 100 c.c. In a known volume of this solution the phosphoric acid, the iron, and the lime are estimated, and the silica in the residue.—*Landw. Versuchstat.*, l., p. 363.

THE COMMERCIAL SEPARATION OF PLATINUM FROM GOLD.

By E. PRIWOZNIK.

Scraps, filings, &c., of gold containing platinum are put on one side in minting operations, as the ordinary methods of gold refining cannot be applied to them with success. The following method has been successfully used by the author when dealing with large quantities of such scrap.

The sifted filings are digested with nitric acid of 1·199 density, until no more silver is dissolved. During this operation a small quantity of platinum also goes into solution in the form of platino-nitrite of silver. The metallic residue is washed and exhausted with aqua regia of the following composition:—

Concentrated hydrochloric acid	100 volumes
,, nitric acid	43 ,,
Water	143 ,,

When the chloride of silver which deposits on the surface of the metal stops all further action, the solution of chloride of gold is decanted, and the chloride of silver removed by washing the metal with a little ammonia. The metal may then be treated a second time with aqua regia.

After six successive treatments with aqua regia and ammonia, the metallic residue consists of pure platinum. The acid solution which contains the gold is evaporated down with an excess of hydrochloric acid, to drive off the nitric acid, until the chloride of gold crystallises. The chloride of gold is re-dissolved, and the small quantity of platinum present is precipitated by means of chloride of ammonium. Finally, the gold is precipitated by ferrous sulphate.

The filings treated by the author contained:—

Gold	28·05 per cent
Silver	10·56 ,,
Platinum	45·46 ,,
Copper	15·93 ,,

If the metals form a true alloy it is preferable to previously melt this alloy with 3 parts of lead, or, what is even better, 3 parts of zinc. The metal is then granulated and treated with sulphuric acid, which dissolves the zinc, and the spongy residue is treated in the manner above described.—*Œsterr. Zeits. f. Berg. u. Hüttenwesen*, 1899, p. 356.

ALINITE.

The preparation known under the name of *alinite* is a yellowish powder containing a pure culture of Caron's *Bacillus Ellenbachensis alpha*. This bacillus has the property of assimilating atmospheric nitrogen, and thus renders the use of nitrated manures for the culture of cereals more or less superfluous.

Stoklasa has shown that this bacillus is a denitrifying one and that it decomposes fibrine, making 22 per cent of its nitrogen soluble after seventy-six days; while in another experiment, without inoculation, only four per cent of the nitrogen passed to the soluble state. Analogous experiments with peat show that in seventy-two days 42 per cent of the nitrogen passed into the soluble state under the influence of this microbe. Finally, experiments made with barley showed that fixation of free nitrogen took place in the presence of alinite.

Stoklasa, however, came to the conclusion that the principal work of the bacillus consisted in the transformation of the nitrogen of the organic matter in the soil into assimilable nitrogen.

Stutzer and Hartleb concluded that the bacillus is aërobic, that is to say, very similar to the *B. Mycoides* and the *B. Megatherium*, and that in its manner of behaviour with nitrated substances it resembles the bacteria of putrefaction.

According to Lauck, alinite would be a pure culture of the *Bacillus subtilis Ehrenberg* developed in a special preparation of powdered potato. He found that this bacillus has the property of liquefying the albumen of a boiled egg. A culture of this bacillus, to which was added some peptone, soon gave off the characteristic smell of trimethylamine.

As for the practical utility of alinite, Caron states that according to his experiments the use of this substance increases the crops. On the other hand, Moercker shows it to be absolutely inefficient.

A large number of experiments have been carried out in the open field, but the results have been very contradictory.—*Biedermann's Centralblatt*, 1899, xxviii., 156.

THE PRODUCTION OF CRYSTALLISED SALTS OF BISMUTH.

By A. de SCHULTEN.

Part I.

Oxychloride of bismuth, BiOCl, has been obtained crystallised in white flakes having a micaceous lustre, by Jacquelain, by passing a current of steam through chloride of bismuth kept in fusion (*Ann. Chim. Phys.*, lxvi., p. 113). I have prepared crystals of this substance by the following process:—

Three grms. of oxide of bismuth are dissolved in 300 c.c. of hydrochloric acid of 1·05 density, after heating to boiling; 2·5 litres of boiling water are added, and the whole filtered; the filtrate is heated until the whole of the precipitate is re-dissolved, and then allowed to cool. After standing for 24 hours crystals of oxychloride of bismuth are formed; these are washed with water acidulated with hydrochloric acid, then with pure water. The analysis of the crystals gives the following figures:—

	Found.	Calculated for BiOCl.
Bi	80·38	80·21
Cl	13·61	13·64
O	6·01 (by diff.)	6·15
	100·00	100·00

When heated to bright redness these crystals melt, forming a crystalline glass, and give off fumes of chloride of bismuth.

Oxychloride of bismuth occurs as clear, colourless, quadratic crystals. The facets p (001) and $b^{\frac{1}{2}}$ (111) are observed. The crystals are uniaxial and negative; their density is 7·717 at 15°; their molecular volume is thus 33·7.

Oxybromide of bismuth, BiOBr, is obtained in the crystalline state in the following manner. We dissolve 3 grms. of oxide of bismuth in 50 c.c, of hydrobromic acid of 1·38 density; heat the solution to boiling, add 1500 to 1600 c.c. of boiling water, and heat until the whole of the precipitate is re-dissolved. On cooling the solution, a small quantity of well crystallised oxybromide of bismuth is formed. The crystals are washed with water, acidulated with hydrobromic acid, and then with pure water. The analysis of the crystals gives the following figures:—

	Found.	Calculated for BiOBr.
Bi	68·43	68·48
Br	26·24	26·26
O	5·33 (by diff.)	5·26
	100·00	100·00

When heated to bright redness, these crystals melt, forming a crystalline glass, and give off fumes of bromide of bismuth.

Oxybromide of bismuth occurs as clear, colourless, quadratic crystals. The same facts are observed as with the oxychloride. The crystals are uniaxial and negative. Their density is 8·082 at 15°; their molecular volume is therefore 37·7.

Oxyiodide of bismuth, BiOI, is obtained in the crystalline state in the following manner: 0·250 grm. of oxide of bismuth is dissolved in 40 c.c. of hydriodic acid of 1·2 density, the solution is diluted with 6 litres of cold water, and the whole heated on the water-bath. As the temperature rises beautiful crystals of oxyiodide of bismuth are formed. These crystals are washed with pure water. In the preparation of oxyiodide of bismuth it is indispensable to use a large excess of hydriodic acid to prevent the formation of black crystals of iodide of bismuth by the ulterior action of the water. The analysis of the crystals gives the following results:—

	Found.	Calculated for BiOI.
Bi	59·44	59·34
I	35·76	36·11
O	4·80 (by diff.)	4·55
	100·00	100·00

When heated to redness these crystals melt with decomposition.

Oxyiodide of bismuth occurs as clear, quadratic crystals having a beautiful copper colour. Their facets are the same as those observed with the corresponding chlorised compound. The crystals are uniaxial and negative. Their density is 7·922, and their molecular volume is therefore 44·4—*Bull. Soc. Chim.*, Series 3, xxiii., No. 5.

ON THE

PRESENT ASPECT OF THE QUESTION OF THE RETROGRESSION OF THE SOLUBLE PHOSPHORIC ACID IN SUPERPHOSPHATES.

By L. SCHUCHT.

Since the use of mineral phosphates in the manufacture of superphosphates, we find ourselves in the presence of questions which had not previously existed in the case of bone and guano phosphates. There was then no motive or considering the question of the retrogression of the soluble phosphoric acid, owing to the absence of the body which caused this reaction. Such phenomena only began to be evident when we became forced to have recourse to mineral phosphates. These phosphates were submitted to the same treatment as those previously used, and it was found before long that the phosphoric acid which had been rendered soluble retrograded, or, in other words, it gradually became insoluble. When once this fault was recognised, efforts were made to find a remedy, and the method of breaking up the mineral phosphates has been modified and improved to such a degree that at the present time superphosphates can be obtained which are in no way inferior to those prepared from pure raw materials.

But it may be asked, what are the bodies which cause the phenomena of retrogressions? Although this question may not yet be quite decided, we know at least what precautions to take when employing any particular class of raw phosphate. It is therefore necessary, before breaking up the raw phosphate, to submit a properly selected sample to a complete qualitative and quantitative analysis. The results obtained enable us to combine the bases with the acids found, to form salts, and thus to calculate the quantity of acid necessary for its decomposition. Once in possession of these facts, it is easy to make a series of disaggregation tests, using a quantity of sulphuric acid slightly higher than the theoretical amount. From these tests we can also find the amount of phosphoric acid which it is possible to make soluble, and also the degree of decomposition of the phosphate under examination.

The yield of a phosphate depends essentially upon its degree of decomposition by sulphuric acid and the nature and amount of foreign bodies it may contain,—in other words, it is necessary to know the proportion of phosphate that escapes decomposition, the nature of the insoluble residue, and the composition of the salts formed. The superphosphates are variable mixtures of salts, acids, and insoluble bodies, which, being able to react one on the other, may give rise to precipitates. We will return to this point later on, and we wish particularly to point out under what conditions a well-prepared superphosphate may undergo retrogression.

Superphosphate kept in a warehouse exists at first in the pulverulent state, without the particles being in intimate contact; but as fresh quantities are added the pile increases in height, and slowly becomes more and more solid, eventually forming a single, more or less homogeneous lump. It is only from this point that the phenomenon of retrogression is made manifest.

The hygroscopic water and the free acid are set at liberty, and complete the intimate contact of the particles till then isolated. The different bodies become, as it were, suspended in a liquid, and from that moment all the requisite conditions exist for them to act one on the other; *corpora non agunt nisi soluta.*

It is evident that in drying the superphosphate at 100° we only accelerate the decomposition, and start reactions going which, with an undried superphosphate, only take place after some time.

Florida phosphate contains about 46 per cent of soluble material — $CaH_4P_2O_8.H_2O$, H_3PO_4, $Fe_2(SO_4)_3.9H_2O$, $Al_2(SO_4)_3.18H_2O$, $MgSO_4.7H_2O$; and about 54 per cent of insoluble matter — undecomposed phosphate, $CaSO_4.2H_2O$, CaH_2, SiO_2, H_2SiO_3, $Al_2(SiO_3)_3$.

What is the relation between the insoluble residue and the soluble materials? The gypsum, which is the skeleton of the superphosphate, is a neutral body; it is not attacked by phosphoric acid, and Berthollet's law does not apply in this case.

Of all the above-mentioned soluble bodies, the free phosphoric acid (H_3PO_4) is the most active, and it attracts, so to speak, each of the insoluble particles to saturate its own chemical acidity. The undecomposed phosphate is attacked first, then comes the silicate, and, if this latter is present in large quantity, the phosphoric acid will not

be sufficient; there is then a loss of soluble phosphoric acid in the form of $AlPO_4$ and $CaHPO_4$. The calcic fluoride is not attacked, and the silica does not come into the question. As for the insoluble phosphoric acid, there is no longer any left in the stored superphosphate in the form of $Ca_3P_2O_4$, but as acid phosphate of lime. With a proportion of 4 per cent of phosphoric acid in the form of H_3PO_4, the superphosphate may contain 2 per cent of phosphoric acid as $Ca_3P_2O_8$, without there being any $CaHPO_4$ formed. Thus, in the warehouse, the phosphate ought to be richer in soluble phosphoric acid, but, on the other hand, the soluble phosphoric acid may form insoluble compounds.

In the second place, we must refer to the relation between the acid phosphate of lime and the salts of iron and aluminium. The ferric oxide in Florida phosphate exists in the state of $FePO_4$ (1 per cent), and immediately on its disaggregation it is entirely dissolved, and, with the phosphate of lime, forms the compound $Fe_2(SO_4)_3.9H_2O$, which is found in the superphosphate. When the ferric oxide present does not exceed 1 per cent, the ferric sulphate remains in solution with the acid phosphate of lime without there being any loss of soluble phosphoric acid; but if the quantity present is greater, we must expect a retrogression of the soluble phosphoric acid, from the moment when the free phosphoric acid alone is not sufficient to stop the formation of $FePO_4$. All the old ideas as to the composition of the precipitated ferric phosphate, $Fe_2O_3(PtO_5)_2$, $2Fe_2O_3(P_2O_5)_3$, &c., are erroneous; there is simply $FePO_4,2H_2O$ or $FePO_4$ formed.

The alumina is in the form $AlPO_4$ in the soluble portion, and in the state of clay Al_2SiO_5 in the gangue; free alumina, Al_2O_3, has only up to the present been found in phosphate from Somme. Phosphate of aluminium is easily decomposed, but the sulphate is not so active on the acid phosphate of lime as ferric sulphate. We may therefore say that ferric oxide is more objectionable than alumina.

The salts of magnesia, as well as the small quantities of alkaline salts which are present in superphosphates, have no influence on the retrogression of the soluble phosphoric acid.—*Die Chemische Industrie*, 1899, p. 152.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MARCH 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, April 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from March 1st to March 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The recent excessive rainfall ended with the last day of February, and was followed by dry weather during the first fourteen days of March. The total rainfall for the month at Oxford has been 0·50 inch, distributed over seven days. The average fall is 1·50 inches, we therefore have a deficit of 1·0 inch of rain for the month; this reduces the excess for the year to 1·61 inch, equal to 29·7 per cent on the thirty years' average.

Our bacteriological examinations of 537 samples have given the results recorded in the following table; we have also examined 37 other samples, from special standpipes, &c., making 574 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	282
New River, filtered (mean of 110 samples) ..	8
Thames, unfiltered (mean of 27 samples) ..	1694
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 304 samples)	18
Ditto ditto .. .. highest	202
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 27 samples) ..	426
River Lea, from the East London Company's clear-water wells (mean of 42 samples) ..	5

The bacteriological purity of the London water supply during the last month has been very good on the whole, there being only one occasion on which one of the filters of the Thames-derived Companies gave a higher number of microbes than 200 per c.c. All the other samples examined were well filtered, the average number of microbes not exceeding 18 in the Thames-derived Companies, and being as low as 5 in the River Lea water from the East London Water Company's mains.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Annual General Meeting, March 29th, 1900.

Professor THORPE, F.R.S., President, in the Chair.

DR. DYER and Mr. FRISWELL were appointed scrutators, and a ballot was opened for the election of Officers and Council for the ensuing year, the ballot being closed at the conclusion of the President's Address.

In beginning his Address, the PRESIDENT congratulated the meeting on the fact that the Society continues to grow and prosper; the number of its Fellows steadily increases, and their communications continue to add to its dignity and usefulness. Its financial position is no less satisfactory.

The numerical strength of the Society was as follows:—

Number of Fellows, March 29th, 1899	2230	
,, ,, since elected	118	
,, ,, reinstated by Council	4	
		2352
Removed on account of non-payment of two annual subscriptions	15	
Withdrawn	20	
Deaths	25	
	—	60
Number of Fellows, March 29th, 1900		2292
Foreign Members		33

The following have died:—Joshua Buchanan, Robert Wilhelm Bunsen, Kanny Lall Dey, J. B. Edwards, Sir Edward Frankland, Charles Friedel, William Harkness, John F. Hodges, John Elias Hughes, Eric H. Jackson, William Marcet, Alexander McDougall, Walter Newton, L. F. Nilson, G. H. Ogston, Robert Oxland, R. T. Plimpton, C. F. Rammelsberg, Henry Charles Reynolds, J. G. F. Richardson, W. H. Richardson, Thomas Glazebrook Rylands, Edward C. Cortis Stanford, Sidney Augustus Sworn, Andrew Taylor, William Thorp, Ferdinand Tiemann, Peter Waage, David Watson, W. Lloyd Williams.

The passing away of so eminent a member of the Society, and of one who played such a leading part in the development of chemistry, as Sir Edward Frankland is no ordinary event in the history both of Science and of the Society, and demands a special reference. The Council have accordingly determined that Sir Edward Frankland's services shall be commemorated in the manner hitherto restricted, with the special exception of the late Professor von Hofmann, to its Honorary Foreign Members; and they have requested his pupil, collaborator, and friend, Professor Armstrong, to give a Memorial Lecture on his life-work in the ensuing autumn.

The names of the deceased Foreign Members are Robert Wilhelm Bunsen, Charles Friedel, Lars Fredrik Nilson, Carl Friedrich Rammelsberg, and Peter Waage.

Bunsen, who was elected a Foreign Member during the first session of the Society, namely, as far back as February 1, 1842, and who was long the *doyen* on the list, died on August 16, 1899, in his eighty-ninth year. His friend and pupil, Sir Henry Roscoe, a past President of the Society, has undertaken, at the request of the Council, to commemorate the signal services which Professor Bunsen rendered to physical science.

Charles Friedel, Member of the Institute and Professor of Chemistry in the Sarbonne, who was elected a Foreign Member of the Society on May 18th, 1876, died on April 20th, 1899, at the age of sixty-seven. His friend and co-worker, Professor J. M. Crafts, of the Massachusetts Institute of Technology, and a Fellow of this Society, has kindly responded to the invitation of the Council to prepare a Memorial Lecture, which he hopes to be able to deliver on some evening in the latter half of June next.

Lars Fredrik Nilson, Professor of Agricultural Chemistry in the Kongl. Landtbruks-Akademien, Stockholm, and formerly Professor of Analytical Chemistry in the University of Upsala, whose name is specially connected with the chemistry of the so-called rare earths, and who was elected a Foreign Member of the Society on February 2, 1888, died on May 14, 1899, in his fifty-ninth year. Professor Otto Pettersson, of the Free University of Stockholm, who was associated with Nilson in most of his work in pure chemistry, has undertaken, in compliance with the wish of the Council, to give the Society an account of the many services which his friend rendered to chemistry, and he hopes to be able to visit London for this purpose in the first week of July next.

Carl Friedrich Rammelsberg, Geh. Reg. Rath. and Emeritus Professor of Inorganic Chemistry in the University of Berlin, who was elected a Foreign Member on May 3, 1866, died on December 28, 1899, in his eighty-seventh year. Professor H. A. Miers, of Magdalen College, Oxford, and a Fellow of this Society, has kindly acceded to the wish of the Council to prepare a memorial lecture on the life-work of the veteran mineralogical chemist, which he hopes to deliver to the Society in December next.

Peter Waage, Professor of Chemistry in the University of Christiania, whose name is best known in connection with the studies on chemical affinity which he made in association with his colleague, Professor Guldberg, died on January 13th of this year. He was elected a Foreign Member of this Society on January 20th, 1898.

Since the last Anniversary, 175 communications have been made to the Society, a number greater than in any preceding year. In character, variety, and importance they compare not unfavourably with the contributions of any former period. Abstracts of all these have appeared in the *Proceedings*, and 83 have already been published in the *Transactions*. The volume of *Transactions* for 1899 contains 120 memoirs, occupying 1166 pages; in the preceding year 102 papers were published, occupying 1038 pages.

The volumes for 1899 contains 3617 abstracts of papers, published mainly in continental journals, occupying 1796 pages, arranged as follows:—

	Pages.	No. of Abstracts.
Part I.		
Organic Chemistry	968	1477
Part II.		
General and Physical Chemistry		421
Inorganic Chemistry		417
Mineralogical Chemistry		211
Physiological Chemistry		236
Chemistry of Vegetable Physiology and Agriculture		244
Analytical Chemistry		611
	828	2140
Total in Parts I. and II. ..	1796	3617

The number of abstracts dealt with in 1899 is 791 more than in the preceding year. All arrears in abstracting have at length been overtaken. The Fellows may now count upon having the monthly *résumé* of contemporary chemical literature brought to their attention as quickly as it is possible to furnish it.

It will have been observed also that the Editor has succeeded in issuing the journal somewhat earlier in the month than has hitherto been thought possible.

The question of co-operation with other English-speaking Chemical Societies in the preparation of abstracts of foreign chemical literature was referred to in the last Presidential Address. But, although the matter has been considered, the committee appointed to deal with this question has not yet reported.

637 copies of the 1883—92 Index, and 317 of the 1873—82 Index were issued to Foreign Members, Institutions, and to those Fellows who, being entitled to a Free Grant of them, had made application within the prescribed period. 565 of these applications had been received, of which 43 were those of Fellows resident abroad.

The work of compilation of the Collective Index showed the necessity of indexing the annual volumes in a more systematic manner than had been done in the past. For the last four years, the work of indexing the annual volumes has been entrusted to a special staff of indexers under the direction of the Sub-Editor. This plan has worked well as regards uniformity and consistency, but unfortunately it has not been possible, by means of it, to bring out the Annual Index with that promptitude which is desirable. The Index Committee has therefore suggested to the Council the propriety of entrusting the work of indexing to one person, to be responsible, under the direction of the Editor, for the preparation and speedy publication of the work, and the Council has sanctioned the employment, on this work, of Mrs. Dougal, who is already favourably known to the Fellows for the excellent service she rendered the Society in editing the Collective Indexes of its publications since 1873.

The Fellows are aware the Society stocks a certain number of its publications, and the question of the custody and supply of back numbers has received attention. A limited number of sets of the *Memoirs* and *Proceedings* of 1841—7, of the *Quarterly Journal*, 1849—62, and of the First Series of the *Journal* from 1863 onwards are now on sale.

As regards the Library, 790 books have been borrowed, as against 727 last year; and 114 books, 397 volumes of periodicals, and 27 pamphlets have been added, as against 67 books, 285 volumes of periodicals, and 24 pamphlets in the preceding year.

The condition of the Library Catalogue has received attention, and the Council has directed a new Catalogue to be prepared according to a scheme drawn up by the Library Committee; and the work is well advanced under the direction of Mr. Steele, assisted by Miss Morfee.

Grants amounting to £192 have been made in aid of chemical investigation.

The Longstaff Medal has been awarded to Professor W. H. Perkin, F.R.S. In making the presentation, the President said :—

"Dr. Perkin, it gives me a special pleasure to ask you, on behalf of the Chemical Society, to transmit this medal to your son, Professor W. H. Perkin, who, to our great regret, is unable to be here to receive it in person. The Longstaff Medal has been awarded to him for his researches on closed chain compounds of the trimethylene and similar series, and for his recent important syntheses of camphor derivatives.

"The Council desire on making this award to indicate how highly we regard the contributions with which he has enriched our *Transactions*. Under his stimulating influence, the chemical laboratory of the Owens College, and especially that section of it under his more immediate direction, continues, as of old, to furnish a succession of valuable memoirs to English chemical literature. We trust that this activity may long be maintained, and that our Society may continue, as in the past, to enjoy the credit and distinction of disseminating the results of the inquiries to which his fruitful ideas give rise."

During the long vacation, the building has been thoroughly cleaned and re-decorated, and certain much needed improvements in the lavatory accommodation and in the sanitary arrangements of the building have been effected.

The accommodation at the disposal of the Society is, however, a matter of growing concern. The meeting-room seats no more than 157 persons, in spite of the increased space which was gained as the results of the alterations in 1892. This is altogether incommensurate with the present numerical strength of the Society, and, as the Fellows well know, considerable inconvenience is occasionally felt owing to the impossibility of finding room for those who wish to attend the meetings. The collection of books is gradually overspreading into every room the Society possesses, and such conveniences as the officers have for the discharge of their official business, and for the custody of the official records, are greatly curtailed by the increasing inadequacy of the space at their disposal.

The Society is indebted to several of its Fellows for additions to its artistic possessions; among these is a bust of Sir Humphry Davy, presented by Dr. Debus, a copy of which in bronze, together with its pedestal, has been given by Dr. Messel; Dr. Atkinson has presented an electrotype medallion of Wöhler, executed by Dr. Hugo Müller, and Sir Henry Roscoe has given some photographic mementoes of his association with Bunsen and the Heidelberg Laboratory.

The Society had the pleasure of congratulating, by a suitable address, Sir George Gabriel Stokes on the occasion of his jubilee as Lucasian Professor of Mathematics, and it has since received from the University of Cambridge an exemplar of the medal which was struck in commemoration of that event.

Congratulations have also been sent to Professor Emil Fischer and Professor van 't Hoff on the twenty-fifth anniversaries of their doctorates, of which acknowledgments, already published in the *Proceedings*, have been received.

The remainder of the address was devoted to a review of the progress of chemical science from the beginning of the century to the date of the formation of the Society.

Sir H. Roscoe, F.R.S., proposed a vote of thanks to the President, coupled with the request that he would allow his Address to be printed in the *Transactions*.

Prof. Liveing, F.R.S., seconded the motion, which was carried by acclamation.

The President having returned thanks,

Prof. Tilden, F.R.S., the Treasurer, in giving an account of the balance sheet, which he laid before the Society, duly audited, said :—

The receipts had been :—By admission fees and subscriptions, £4088; by sale of Journal and advertisements, £781 15s. 6d.; and by dividends on invested capital, £444 13s. 7d. The expenses had been :—On account of the Journal, £3388 12s. 11d.; on account of the Proceedings, £171 14s. 7d.; on account of the preparation of a new Card Catalogue, £35 9s. 2d.; on account of the Library, £286 17s. 3d.; House expenses, including the re-decoration of the building, £543 5s. 11d.; the total expenditure being £4993 13s. 1d. Grants amounting to £192 had been made to Fellows from the Research Fund during the year.

Attention was directed to the fact that the net income of the Society for the year being £5371 1s. 5d., and the expenses £4993 13s. 1d., the surplus is only £377 8s. 4d. This is considerably less than the amount, £578, of the Composition and Admission Fees which ought to be regarded as Capital. In view of the steadily increasing cost of the Society's publications, the recurrence of the heavy charge for the Decennial Index, and the growth of the Library, economy will have to be practised to keep the finances of the Society in a healthy condition. The Treasurer appealed to authors of papers to assist in keeping down the cost of the composing and correcting the proofs of papers communicated to the Society. At the present time the average cost of corrections amounts to more than one-third the charges for putting manuscript into type. A little more care exercised by authors in preparing for the press would result in a substantial reduction in the cost of printing.

The Treasurer, in concluding, proposed a vote of thanks to the auditors, which was acknowledged by Mr. Page.

Prof. Odling, F.R.S., proposed that the thanks of the Fellows be tendered to the Treasurer for his services during the past year; this motion was seconded by Dr. Russell, F.R.S., and carried.

Prof. H. B. Dixon, F.R.S., proposed a vote of thanks to the Officers and Council.

Prof. Warington, F.R.S., seconded the motion, which was unanimously adopted.

Prof. Dunstan, F.R.S., responded on behalf of the Council.

Prof. Dewar, F.R.S., proposed a vote of thanks to the Editor, Sub-Editor, Abstractors, and Indexers, which was seconded by Mr. Groves, F.R.S., and carried.

Dr. Wynne, F.R.S., responded.

The scrutators having presented their report to the President, he declared that the following had been duly elected :—

President — T. E. Thorpe, Ph.D., D.Sc., LL.D., For.Sec.R.S.

Vice-Presidents who have filled the office of President— Sir F. A. Abel, Bart., K.C.B., D.C.L., F.R.S.; H. E. Armstrong, Ph.D., LL.D., F.R.S.; A. Crum Brown, D.Sc., LL.D., F.R.S.; Sir W. Crookes, F.R.S.; James Dewar, M.A., LL.D., F.R.S.; Sir J. H. Gilbert, Ph.D., LL.D., F.R.S.; J. H. Gladstone, Ph.D., D.Sc., F.R.S.; A. Vernon Harcourt, M.A., D.C.L., F.R.S.; H. Müller, Ph.D., LL.D., F.R.S.; W. Odling, M.B., F.R.S.; W. H. Perkin, LL.D., Ph.D., F.R.S.; Sir H. E. Roscoe, D.C.L., LL.D., F.R.S.; W. J. Russell, Ph.D., F.R.S.; A. W. Williamson, LL.D., F.R.S.

Vice-Presidents—E. Divers, M.D., D.Sc., F.R.S.; C. E. Groves, F.R.S.; G. D. Liveing, M.A., D.Sc., F.R.S.; T. Purdie, Ph.D., F.R.S.; T. Stevenson, M.D.; John M. Thomson, LL.D., F.R.S.

Secretaries—Wyndham R. Dunstan, M.A., F.R.S.; A. Scott, M.A., D.Sc., F.R.S.
Foreign Secretary—Raphael Meldola, F.R.S.
Treasurer—William A. Tilden, D.Sc., F.R.S.
Other Members of Council—H. Brereton Baker, M.A.; F. D. Chattaway, Ph.D., D.Sc.; Frank Clowes, D.Sc.; J. Norman Collie, Ph.D., F.R.S.; A. E. Dixon, M.D.; H. J. H. Fenton, M.A., F.R.S.; W. Gowland; C. T. Heycock, M.A., F.R.S.; D. Howard; Rudolph Messel, Ph.D.; W. J. Pope; James Walker, D.Sc.

Extra Meeting, March 29th, 1900.

Professor Thorpe, F.R.S., President, in the Chair.

Sir Henry E. Roscoe, F.R.S., Vice-President, delivered the Bunsen Memorial Lecture.

Before commencing the Lecture, Sir Henry Roscoe read the following telegram, which he had just received from Bunsen's nephew and executor, Dr. Philipp Bunsen, of Marburg:—"On the occasion of the Memorial Lecture, the Bunsen family joins sincerely with the illustrious Society, and sends respectful thanks and compliments."

The death of Bunsen on August 16th, 1899, severs the last link connecting the chemists of our time with the great men of the earlier part of the century, Berzelius, Gay Lussac, Dumas, Liebig, and Wöhler, Mitscherlich, and the two Roses, as well as with the physicists Dove, Wilhelm Weber, and Magnus, all of whom he counted amongst his personal friends. Living to the ripe age of eighty-eight years, he was destined to witness the deaths as well as the scientific births of many distinguished colleagues and pupils; of Kirchhoff, Helmholtz, Kopp, and Hermann; of Strecker, Kolbe, Kekulé, Pebal, Lothar Meyer, and, finally, of Victor Meyer, his successor in the chair at Heidelberg.

Robert Wilhelm Bunsen was born on March 31st, 1811, at Göttingen, where his father was chief University Librarian, and Professor of Modern Philology. Entering the University in 1828, he studied chemistry under Stromeyer, and graduated in 1830. He then visited Paris, Berlin, and Vienna, continuing his studies, and making the acquaintance of the scientific men of those cities, returning to Göttingen, where, in 1834, he was admitted by the University as Privatdozent in Chemistry. In 1836 he succeeded Wöhler as teacher of chemistry in the Polytechnic School at Cassel, and in 1839 was appointed Professor Extraordinarius of Chemistry at Marburg, being advanced in 1842 to the post of Ordinarius. Here he remained till 1851, when he went for a short period to Breslau, and in 1852 he accepted the chair at Heidelberg, which he occupied until his retirement in 1889. In these several positions, Bunsen laboured incessantly and devotedly for fifty-six years in the furtherance of chemical science, with the result that his name will be handed down to posterity as one whose work has earned for him the very first rank amongst chemists of the nineteenth century.

Bunsen's first published paper recorded his discovery that freshly precipitated ferric hydroxide acted as a powerful antidote in cases of arsenical poisoning, but the first research in which he showed his power was the classical one on the cacodyl compounds, which was continued for no less than six years. His important investigations on the composition of the gases of German and English blast-furnaces were of the highest practical as well as theoretical importance, and led incidentally to the devising and perfecting of his well-known system of gas analysis. In 1857, he collected in a volume the whole of his gasometric researches, and of this—the only book he ever published—a second and greatly enlarged edition appeared in 1877.

The construction of his carbon-zinc battery marks an era in the economic production of electricity. He showed how it could be employed as a means of illumination; he determined its power of doing work, and used it to prepare electrolytically many metals hitherto unknown in a coherent form, notably magnesium, the preparation of which had baffled Davy. These researches led in turn to his inventions of the well-known "grease spot" photometer and the ice- and the vapour-calorimeters. Along with the lecturer, he carried out the photochemical researches based on the action of light on a mixture of equal volumes of hydrogen and chlorine, pronounced by Ostwald to be "the classical example for all further researches in physical chemistry."

The famous researches on spectrum analysis of Kirchhoff and Bunsen gave, not only a full explanation of Frauenhofer's lines in the solar spectrum and a knowledge of the composition of the sun and the fixed stars, but led in Bunsen's hands to the discovery of two new metals, cæsium and rubidium, and in the hands of others, of thallium, indium, gallium, and other elements.

In 1846, Bunsen spent three months and a half in Iceland, as a member of an expedition promoted by the Danish Government, of which the outcome was his well-known series of investigations of the volcanic and pseudo-volcanic phenomena of that island. He worked for some time at the separation of the metals in the residues left after the extraction of the platinum, as carried out in the Imperial Mint at St. Petersburg, and in his account of this is to be found a description of his filter-pump actuated by a fall of water.

To Bunsen and Schischkoff we owe the first exact examination both of the gaseous and solid products of the explosion of gunpowder. Perhaps his name is most widely known in connection with his burner for gas, devised to provide a flame both hotter and more steady than that of the argand burner with wire-gauze top previously in use. Not only as a source of heat, but also most ingeniously as an analytic agent, was the flame of his gas burner employed in producing the delicate and characteristic "flame reactions." Important also are his papers on the colour of water, iodometric methods, and water analysis.

The remainder of the Lecture consisted of the lecturer's personal recollections of Bunsen, and of the Heidelberg Laboratories from 1852 onwards, with a description of the character and methods of work of the Master as a teacher and a lecturer, and of the relations existing between him and his pupils.

Considerate and generous towards the opinions of others, he held firmly to his own, which at times he did not fail strongly to express; simple and straightforward, he disliked assumption and hated duplicity; single-minded and wholly devoted to his science, he abhorred vanity and despised popularity hunting.

At the time of his death, Bunsen had been for many years our senior Foreign Member, having been elected on February 1, 1842, during the first session of our Society. In 1858 he became a Foreign Fellow of the Royal Society, and in 1860 the Copley Medal was awarded to him. To Bunsen and Kirchhoff, in 1877, the first award of the Davy Medal was made in recognition of their researches and discoveries in spectrum analysis.

Another English honour conferred upon him was that of the award in 1898 of the Albert Medal of the Society of Arts, given for "distinguished merit in promoting art, manufactures, or commerce," in recognition of his numerous and most valuable applications of chemistry and physics to the arts and to manufactures.

On the motion of Professor Odling, seconded by Dr. E. Atkinson, a vote of thanks was passed to Sir Henry Roscoe for his Lecture.

Ordinary Meeting, April 5th, 1900.

Prof. J. M. Thomson, F.R.S., Vice-President, in the Chair.

Certificates were read for the first time in favour of Messrs. James Ashton, Land Hill House, Hoscar Moss,

Ormskirk, Lancs.; Richard Henry Cyril Gompertz, 110, Elgin Crescent, Notting Hill, W.; Alexander Ernest McKenzie, 11, Bank Road, Workington; Hugh McNair, Bellfield, Wishaw; Herbert Procter Smith, 60, Stepney Street, Llanelly; George Edward Tomlins, 4, Radford Road, Hither Green, London, S.E.

Of the following papers, those marked * were read:—

*53. "*The Liquefaction of a Gas by 'Self-cooling.'*" A Lecture Experiment." By G. S. NEWTH.

The principle of the modern method for obtaining liquid air or oxygen by the "regenerative" method may be illustrated for lecture purposes by means of nitrous oxide, the spiral of fine copper tube being attached directly to the steel bottle containing nitrous oxide, and inserted into a Dewar vacuum-jacketed test-tube. No other insulating arrangements are required. In order to maintain the necessary pressure in the nitrous oxide bottle the latter is kept in warm water at a temperature of about 30°.

*54. "*Note on Partially Miscible Aqueous Inorganic Solutions.*" By G. S. NEWTH.

When strong aqueous ammonia (sp. gr. 0·880) is added to a saturated solution of potassium carbonate and the liquids shaken together, two distinct layers separate out, just as in the case of water and ether. At ordinary temperatures, it is found that the potassium carbonate solution dissolves about 37 per cent of the ammonia solution, while the latter dissolves about 6 per cent of the potassium carbonate solution by volume.

The solubility of each liquid in the other increases with rise of temperature, the curves of their solubilities intersecting at 43°, above which temperature the solutions are miscible in all proportions. A small amount of added water is also found to increase the mutual solubility; with 12·9 per cent of added water, the separate phases cease to exist above 10°, whilst if 18·1 per cent of water be added, the liquids are miscible in all proportions at all temperatures above 0°. When cooled below these critical temperatures, the clear solution instantly becomes turbid by the separation into two liquids. No other aqueous solutions of inorganic substances have as yet been found which exhibit a similar behaviour, but strong aqueous solutions of trimethylamine and triethylamine behave in a like manner with a saturated solution of potassium carbonate.

DISCUSSION.

Dr. DIVERS said that Mr. Newth's paper was interesting on account of the evidence it afforded that we had in aqueous ammonia an analogue of aqueous alcohol, the liquefied ammonia precipitating salts from aqueous solution and becoming dehydrated by potassium carbonate. He had himself shown that ammonium nitrate deliquesced in ammonia gas just as other salts deliquesced in aqueous vapour, and then furnished liquid ammonia under atmospheric pressure, which behaved as a solvent like water or alcohol.

*55. "*The Decomposition of Chlorates.* Part II. *Lead Chlorate.*" By WILLIAM H. SODEAU, B.Sc.

The preparation of pure anhydrous lead chlorate and the examination of the products of its slow decomposition were first dealt with. The solid residue was found to absorb chlorine very readily at the temperature of the decomposition (about 200—240°), hence much of the chlorine first evolved is usually re-absorbed.

When quantities of 1 grm. were decomposed at atmospheric pressure in times varying from fifteen to sixty-five minutes, the chlorine given off varied from 45 to 60·6 per cent of the amount originally present in the chlorate actually decomposed. The employment of a stream of nitrogen increased this proportion, the series of experiments thus carried out pointing to 87·5 per cent of chlorine as the limiting value when re-absorption is entirely eliminated, although Spring and Prost (*Bull. Soc. Chim.*, 1889, [iii], i., 340), obtained only 8 per cent in the decpmposition of large quantities. Only 81·4 per cent could be obtained by reducing the pressure to 2 m.m., but this is probably accounted for by the necessary increase of temperature having facilitated re-absorption; it seems remarkable that decrease of pressure should markedly impede the decomposition of the chlorate. Addition of lead chloride appears to have no effect whatever.

In violent decompositions (lasting about one second) at atmospheric pressure, some oxychloride was formed, and only about 39 per cent of the chlorine was obtained. Similar decompositions at pressures varying from 6 to 20 m.m. gave 58·5 per cent, but the much higher temperature must no doubt have prevented the re-absorption from being even then successfully eliminated.

The author concludes that the slow decomposition of lead chlorate consists of two independent reactions: (1) $Pb(ClO_3)_2 = PbCl_2 + 3O_2$; (2) $Pb(ClO_3)_2 = PbO_2 + Cl_2 + 2O_2$, the latter having about seven times the velocity of the former. At the same time, the reaction $PbO_2 + Cl_2 = PbCl_2 + O_2$ proceeds to a greater or less extent according to the conditions, and may result in much of the chlorine being re-absorbed. The mechanism in rapid decomposition is probably similar, except that the higher temperature decomposes much of the peroxide giving oxychloride.

*56. "*The Bromination of Benzeneazophenol.*" By J. T. HEWITT and W. G. ASTON.

One of the authors recently (*Trans.*, 1900, lxxvii., 99), described the nitration of benzeneazophenol with dilute nitric acid at 40—45°. The production of benzeneazo-*o*-nitrophenol agree with the formulation of benzeneazophenol as a hydroxylic derivative of azobenzene, but was not compatible with the structure of quinonephenylhydrazone. Shortly after the publication of these results, a note by Professor Armstrong (*Proc.*, 1899, xv., 243), appeared in which it was stated that bromine and benzeneazophenol yielded a product giving *p*-bromoaniline and phenol on reduction. The authors find, however, that the first product of the action of bromine on benzeneazophenol is benzeneazo-*o*-dibromophenol, if the bromination is carried out in acetic acid solution to which sodium acetate is added in order to prevent any formation of quinonephenylhydrazone hydrobromide. In fact, with the exercise of care in the matter of cooling, and the slow addition of the acetic acid solution of bromine, a nearly quantitative yield may be obtained, the substance separating immediately in a very pure condition.

Benzeneazo-*o*-dibromophenol melts at 136° (corr.); it forms a well crystallised sodium salt, but no hydrochloride or hydrate has been obtained, although it gives a sulphate with concentrated sulphuric acid.

The *acetyl* derivative melts at 143°, the *benzoyl* derivative at 120°, and the *ethyl ether* at 71°. The latter compound does not show any mobility of its bromine atoms, boiling for one hour with acetone leaving it unchanged. The substitution of other azophenols is being investigated.

*57. "*A New Glucoside from Willow Bark.*" By H. A. D. JOWETT, D.Sc.

Having had occasion to examine a bark purchased as that of black willow, which could not be further identified than as some species of *Salix*, it was found that the glucoside contained in the bark was not salicin, but a new compound. Chemical examination has shown it to be the glucoside of *m*-oxybenzaldehyde, and the name *salinigrin* is provisionally suggested for it. It is contained in the bark to the extent of about 1 per cent, and is a white, crystalline substance melting at 195° (corr.).

It is soluble in 52·2 parts of water and in 218·2 parts of alcohol at 15°, and is lævorotatory $[\alpha]_D$ 15 = −87·3°. From salicin, it is sharply distinguished by yielding a colourless solution with sulphuric acid, salicin under similar conditions producing a blood-red colour. Its formula is $C_{13}H_{16}O_7$, and it splits up, on hydrolysis, into *d*-glucose and *m*-oxybenzaldehyde.

*58. "*Alkylation by Means of Dry Silver Oxide, and Alkyl Iodides.*" By GEORGE DRUCE LANDER, D.Sc.

In addition to the results given (*Proc.*, 1900, 6), the following were described: By the action of dry silver oxide, and ethyl iodide on acetanilide, ethyl *i*-acetanilide, $C_6H_5N{:}C(OC_2H_5)CH_3$, is produced. This is a colourless, mobile liquid, boiling at 207—208°, easily hydrolysed by mineral acids into aniline, acetic acid, and alcohol. By warming with aniline, diphenyl ethenyl amidine, $PhN{:}C(NH{\cdot}Ph)CH_3$ (m. p. 131—132°) is formed.

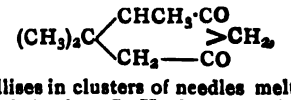
Ethyl malonate, by prolonged boiling with an excess of oxide and ethyl iodide, is partially ethylated, nearly 8 grms. of ethyl ethylmalonate being obtained from 32 grms. of ethyl malonate.

The action of silver oxide and methyl iodide on salicylic acid gave methyl methoxybenzoate boiling at 245°; the free acid melted at 99°.

Oxidation may also occur, as in the following cases: In alkylating benzoin, part is oxidised to benzaldehyde and benzoic acid, the latter being then alkylated. In the alkylation of ethyl acetoacetate, if the iodide be added to the mixed ethereal salt and oxide, a small quantity of a high boiling oxidation product, probably ethyl diacetylfumarate, is formed, which crystallises from ether in fine needles (m. p. 102—105°).

59. "*The Interaction of Mesityl Oxide and Ethyl Sodiomethylmalonate.*" By ARTHUR WILLIAM CROSSLEY.

By the condensation of mesityl oxide with ethyl sodiomethylmalonate, *ethyl trimethyldihydroresorcylate*, $C_{12}H_{18}O_4$, is obtained, which separates from a mixture of chloroform and light petroleum as a microcrystalline powder melting at 94°. On hydrolysis with alcoholic potash, it yields *trimethyldihydroresorcinol*,—



which crystallises in clusters of needles melting at 99·5—100°. The *ethyl* ether, $C_{11}H_{18}O_2$, is a faintly yellow oil boiling at 264—265° (750 m.m.), and the *silver* derivative a white, flocculent precipitate.

By oxidising the resorcinol with sodium hypobromite, it is converted into an acid having the formula $C_8H_{14}O_4$, which melts at 88°, and seems to be *αββ*-trimethylglutaric acid. If the oxidation be arrested before it is complete, a *dibromo-* and a *monobromo*-derivative may be obtained. The former, having the formula $C_9H_{12}Br_2O_2$, crystallises from alcohol in needles melting at 112·5°, and the latter $C_9H_{13}BrO_2$, separates from benzene in glistening plates melting with decomposition at 152°.

The more complete investigation of the properties and reactions of the resorcinol and its derivatives is in progress.

60. "*The Products of the Action of Fused Potash on Dihydroxystearic Acid.*" By HENRY RONDEL LE SUEUR.

From the products resulting from the fusion of dihydroxystearic acid (m. p. 130·5—131°) with potassium hydroxide at a low temperature, two acids have been isolated. One of these is a dibasic acid, $C_{18}H_{34}O_5$, which crystallises from acetic acid in aggregates of long, flat needles melting at 111—111·5°. The *silver* salt, $C_{18}H_{32}O_5Ag_2$, is a white, curdy precipitate, the *calcium* salt crystallises with $3H_2O$, and the *ethyl* salt, $C_{18}H_{32}O_5Et_2$, is a slightly yellow, oily liquid boiling at 269—270° under 30 m.m. pressure. The other acid, $C_{18}H_{34}O_3$, crystallises from dilute alcohol in small plates melting at 78·5—79°, and is probably identical with the acid which Saytzeff (*Journ. Prakt. Chem.*, 1886, xxxiii., 300), obtained in an impure state by the distillation of dihydroxystearic acid.

If the fusion of the dihydroxystearic acid be carried out at a higher temperature, the main products formed are azelaic acid, pelargonic acid, and an oil which boils at 280—300° under 50 m.m. pressure.

The author intends to investigate thoroughly the various products obtained by fusing the dihydroxystearic acids from oleic and elaidic acids) with alkalis, and to study generally the action of fused alkalis on hydroxylated fatty acids.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 14, April 2, 1900.

A New Gaseous Body: Sulphur Perfluoride, SF_6.—H. Moissan and P. Lebeau.—When a small piece of sulphur, supported by a fine platinum wire, is pure in an atmosphere of fluorine above mercury it takes fire, burning with a livid flame, and the mercury rises in the tube. The resulting gas is found to be not absorbed by water and partially absorbed by potash solution. The gas remaining after treatment with alkalis is very stable. These preliminary experiments show that two substances have been formed:—(1) A gaseous body, which is unattacked by water, but absorbed by potash; (2) a gaseous body, unabsorbed by water and alkalis, but able to be destroyed by sodium vapour. Further examination shows that these two sulphur fluorides are always formed together, but, by varying the proportions of sulphur and fluorine, a gas containing 90 per cent of the perfluoride (the portion unattacked by alkalis) is formed. The authors examine the properties of this mixture.

Action of Hydrogen on Antimony Sulphide.—H. Pélabon.—At temperatures above 360°, hydrogen acts on crystallised antimony sulphide, giving sulphuretted hydrogen and antimony. Under the same conditions of temperature, sulphuretted hydrogen attacks antimony, forming the trisulphide, Sb_2S_3. If the operations take place in sealed tubes, the two inverse reactions tend to a system of four bodies in chemical equilibrium. A study of this system leads to the conclusion that the limiting composition of the gaseous mixture depends only on the temperature. The value of ρ increases with the temperature, and this is in perfect harmony with the law of the displacement of equilibrium by temperature, if it is admitted that, under the conditions of the experiment, the reaction $Sb_2S_3+3H_2=3H_2S+2Sb$ is, as at 15°, accompanied by an absorption of heat.

Nickel Arsenide.—Albert Granger and Gaston Didier.—A nickel arsenide, Ni_3As_2, intermediate between those of the two sub-arsenides (Ni_2S, described by Gehlen and Berthier, and natural arsenide, NiAs), was prepared by treating pure nickel with arsenic chloride.

Tungsten Biphosphide.—Ed. Defacoz.—The action of dry gaseous hydrogen phosphide on tungsten hexachloride at a temperature of about 450°, allows of the preparation of a definite new compound—the biphosphide, WP_2. The author studies the properties of this body, and from it prepares the following compounds:—A chloro-phosphide, a double phosphide, and another simple phosphide.

A New Terpenic Alcohol and its Derivatives.—P. Genvresse.—Pinene, under the influence of nitrous vapours or nitrogen peroxide, gives a secondary alcohol, pinenol, which is characterised by its acetic ether, its ketone, and the oxime of this latter. The author has not yet assigned a definite formula for this new body.

Action of Isocyanate of Phenyl and Isothiocyanate of Phenyl on Bibasic Acids.—Elophe Bénech.—It is known that phenyl isocyanate acts on succinic, glutaric, and camphoric acids. M. Haller first obtained the anhydrides of the acids, then the phenylsuccinimide, on one hand, and the glutaric and camphoric dianilides on the other. These derivatives, heated to a high temperature, yield the corresponding imides. The author attempts to generalise this reaction, so as to deduce a general process for the preparation of dianilides corresponding to bibasic acids.

Journal de Pharmacie et de Chimie, Series 6, Vol. xi., No. 1.

A New Method for the Acidimetric Estimation of Vinegar.—M. Durieu.—The following method has given good results with coloured vinegars. Two solutions are prepared:—1. Carbonate of soda, at 5 per cent. 2. Glacial acetic acid, at 7 per cent. The apparatus required is:—A tube gratuated in tenths of a c.c., as used in Esbach's method for the estimation of urea, and a 1 c.c. pipette. 6 c.c. of the alkaline solution are first poured out, so as to have an excess of bicarbonate; then 6 c.c. of alcohol at 95°, the liquids not being mixed; we then add 1 c.c. of the acetic liquor; the total volume then measures twelve divisions; close the tube with the thumb, and shake. When the reaction is finished, the tube is placed in a vessel full of water, and shaken as in estimating urea; the second reading is 19·5, for example. A similar operation is effected with the vinegar under examination, after making quite certain that it does not contain any acetic acid, and a second reading is obtained, say 18·4; by a simple calculation the proportion of acid is found to be 59·70 grms. per litre. The advantages of this method are that no new apparatus is required, and it is of great sensitiveness; it is, however, of great importance that the liquids be accurately measured, and, above all, the the alcohol must not be replaced by water.

No. 2.

This number contains no matter of chemical interest.

No. 3.

Action of Iodine on Antipyrine.—J. Bougault.—The author has already described a method for the estimation of antipyrine by iodine; the present paper has for its object the applications of this method and the examination of the processes of the reaction. The method can be used for the estimation of hypnol and salipyrine, exactly in the same way as for antipyrine; it suffices to remember that 1 grm. of hypnol corresponds to an absorption of 0·7185 grm. of iodine, and 1 grm. of salipyrine to 0·7791 grm. of iodine. It cannot, however, be used for all the compounds of antipyrine. The compound of antipyrine formed during the reaction is monoiodantipyrine, which again forms very complex compounds with the mercuric salts. When we act molecule for molecule with iodine on antipyrine, in the presence of mercuric sublimate in alcoholic solution, it appears that the antipyrine first fixes the iodine, forming an unstable product of addition, which then loses 1 molecule of HI, giving monoiodantipyrine; thus this HI would decompose a corresponding quantity of $HgCl_2$, giving HgI_2 and HCl.

On Iodantipyrine, $C_{11}H_{11}IN_2O$. — J. Bougault. — Iodantipyrine is obtained pure from the compound $(C_{11}H_{11}IN_2O)_4HgI_2HgCl_22HCl$, which is treated with a solution of iodide of potassium made alkaline with carbonate of sodium; the substance is dissociated, the HgI_2, $HgCl_2$, and HCl passing into solution, and the insoluble iodantipyrine remaining. Estimation of the I confirmed the identity of this body:—

	I.	II.	Theory for $C_{11}H_{11}IN_2O$.
Iodine =	40·45	40·63	40·44

The solubility is 0·080 grm. in 100 c.c. of water. Iodantipyrine can also be prepared by the action of iodine on antipyrine in aqueous solution in the presence of acetate of soda; the return is nearly theoretical.

The Spontaneous Formation of Crystallised Oxalate of Manganese during the Permanganic Oxidation of Citric Acid.—G. Denigès.—Ninety grms. of permanganate of potash are dissolved in 3 litres of warm water. The solution is cooled to 15—16°. We also dissolve 300 grms. of citric acid in 600 grms. of warm water, and this solution—also cooled to 15 to 16°—is added to the first, and placed in a beaker surrounded with water at 10—12°. In one or two minutes the mass changes colour, becoming first of a yellowish-red, then brown, and at the same time giving off CO_2 in abundance, while the temperature rises to 30° or 35°. When the gas ceases to come off, the temperature is brought down to 25—28°, and the mixture left alone. After four or five days the sides of the glass become covered with small prismatic crystals, some attaining ½ c.m. in length. The mother-liquor, which is decanted, and which previously contained a very large quantity of acetone-dicarbonic acid, no longer contains a trace. The washed and dried crrystals are rarely homogeneous; the largest ones, rose-coloured and prismatic, can be easily picked out; others appear as small, white, hexagonal crystals, but both kinds have all the analytical reactions of the oxalates. The prismatic crystals gave the following results:—

	Found.	Calculated for $C_2O_4Mn+3aq$.
H_2O (at 110°)	26·90 p.c.	27·41 p.c.
Mn (as Mn_3O_4)	38·95 „	38·76 „
C	12·15 „	12·24 „

And the white crystals gave—

	Found.	Calculated for $C_2O_4Mn+2aq$.
H_2O (at 110°)	20·21 p.c.	20·11 p.c.
Mn (as Mn_3O_4)	42·59 „	42·66 „
C	13·30 „	13·41 „

MISCELLANEOUS.

Popular Chemistry.—The following paragraph is taken from a recent number of a popular contemporary:—"Gold, silver, steel, aluminium, and lead, when immersed in tauric acid, a new chemical discovery, becomes as pliable and ductile as putty."

The Chemists' Exhibition, 1900.—This exhibition, organised by *The British and Colonial Druggist*, will be held at Manchester on June 19th to 22nd. In addition to numerous Pharmaceutical Exhibits, the undertaking will provide examples of the progress made in Dentistry, Optics, Photography, Dietetics, Sanitation, &c. The Chemists' Exhibition is in future to be held alternately in London and the provinces; Manchester is one of the most important centres, as within a radius of a few miles round Manchester is included a population exceeding that of Greater London. Further information relative to the Exhibition can be obtained from the Managing Director, 44, Bishopsgate Without, London, E.C.; personal inquiries can be made any day between 11 a.m. and 12 noon.

Harmful Action of the Fumes of Chloride of Nitrogen.—W. Hentschel.—Accidents due to the inspiration of the fumes of chloride of nitrogen, which hardly appears to be an irritant, have not up to the present been recorded. The author, however, after having been working with solutions of NCl_3 for about six months, was suddenly attacked with inflammation and acute irritation of the mucous membrane, pains in the bronchial tubes, loss of voice, and fever; recovery was very slow. —*Berichte*, xxxii., p. 1878.

On the Density of Liquefied Gases.—A. Ladenburg and C. Krügel.—The authors have already given measurements of the densities of liquefied gases (*Berichte*, xxii., p. 46) by weighing a mass of glass in the hydrostatic balance. Prof. Dewar having made similar experiments by weighing metallic masses, they have repeated their measurements by weighing a mass of silver. In this manner they obtained figures about 3 per cent lower than those previously found,—in fact, very close to those found by Prof. Dewar. They found the density of liquid oxygen, D = 1·134. For liquid air containing x per cent of oxygen, they found $D = 0·86 + 0·00289\,x$. The density of liquid ethylene is 0·6095. They at first thought

that the disagreement between the new and the old results was caused by an inexact determination of the coefficient of expansion of the glass or of the silver at very low temperatures; it has, however, been verified that this is not the case, so that at present the discrepancy cannot be satisfactorily explained.—*Berichte*, xxxii., p. 1415.

Peroxidised Compounds.—P. Melikoff and L. Pissarjevsky.—Theoretical considerations of the peracids and peroxides have led to the following conclusions:—1. The peracids are compounds of the type H_2O_3. When treated with concentrated SO_4H_2, they give off ozone like the peroxides. 2. The compounds of the peroxides with H_2O_2 are entirely comparable with the hydrates of the oxides. 3. These compounds, brought into contact with the peracids give, by double decomposition, water and salts of the peracids and peroxides.—*Journ. Soc. Phys. Chim. R.*, xxxi., p. 273.

MEETINGS FOR THE WEEK.

TUESDAY, May 1st.—Royal Institution, 3. "Studies in British Geography," by Hugh Robert Mill, D.Sc., LL.D., F.R.S.E.

— Royal Institution, 5. Annual Meeting.

WEDNESDAY, 2nd.—Society of Arts, 8. "Some Unfamiliar Masterpieces of the Italian School," by Miss Halsey.

THURSDAY, 3rd.—Royal Institution, 3. "A Century of Chemistry in the Royal Institution," by Prof. Dewar, M.A., LL.D., F.R.S.

— Chemical, 8. "Brazilin" (Part IV.), by A. W. Gilbody, W. H. Perkin, jun., and J. Yates. "Hæmatoxylin" (Part V.), by W. H. Perkin, jun., and J. Yates. "The Substituted Nitrogen Chlorides and Bromides derived from *o*- and *p*-Acet-toluide, and their Relation to the Substitution of Halogens in Toluides and Toluidines," by F. D. Chattaway and K. J. P. Orton.

FRIDAY, 4th.—Royal Institution, 9. "Pottery and Plumbism," by Professor T. E. Thorpe, LL.D., D.Sc., Ph.D., &c.

SATURDAY, 5th.—Royal Institution, 3. "Egypt in the Middle Ages," by Prof. Stanley Lane-Poole, M.A.

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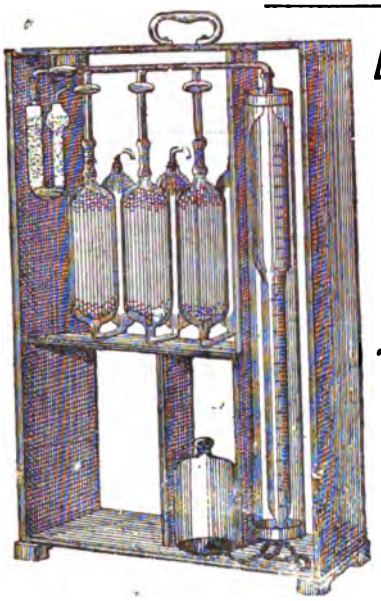

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AFTER much work with liquid air, and many researches on the measurement of low temperatures and the separation of bodies by this means, we consider we are in a position to solve a few questions which have been raised by the splendid researches and discoveries of Ramsay and his co-workers. It seemed to us possible to obtain krypton, of which only little was known at the time we began these labours, in a state of greater purity, and to settle somewhat more accurately its properties, specially also to prove if its atomic weight really amounts, as Ramsay assumes, to 80—82.

The beginning of this work goes back more than a year, as the first large quantity of liquid air which we could obtain (in March of the past year) was lost in transport, and we were only placed in the position to replace this last autumn. In the meantime Ramsay has also worked up larger quantities of liquid air for krypton, as we are informed by the report which appeared in the *Chemiker Zeitung* of the Munich Congress of Physicists. Still there has appeared no authentic publication on krypton (at least known to us) since the first publication in the *Comptes Rendus* (see also *Zeitschrift fur Physikalische Chemie*, xxvi., 362) and the Address published by Ramsay in the German Chemical Society (*Deutschen Chemischen Gesellschaft*, December, 1898), so that we saw no ground for interrupting the work once begun.

After we had learned that the factory Rhenania, near Aachen, had erected a Linde's air-liquefying machine capable of furnishing 50 litres of liquid air per hour, we entered into negotiation with them, and we were permitted to utilise for our purpose the residue of evaporation from 1000 litres of liquid air. Unfortunately there occurred many interruptions in the management of the machinery, so that it was three weeks before a quantity of 850 litres liquid air was gained, of which one of us (K.) brought here as much as 3 litres of the evaporated residue in two of Dewar's flasks, where we collected the gas as it evaporated in large gasometers. We obtained 2300 litres of gas, which were freed from oxygen by passing over red-hot copper. From the remaining 120 litres the nitrogen was separated by essentially the methods mentioned by Lord Rayleigh and Ramsay for the isolation of argon. The last portions of the nitrogen were always oxidised by sparking with surplus oxygen over soda-lye, and the remaining oxygen was absorbed with pyrogallol and soda. In this way a nearly complete separation of the oxygen was obtained, as when treated with yellow phosphorus only a very slight absorption was recognisable.

For sparking over soda-lye we have had made glass vessels of the form shown in Fig. 1, which have answered very well.

From these vessels the gas, after being dried with anhydrous phosphoric acid, was collected into several of Ramsay's gasometers.† In such operations it is self-evident that all intermediate parts must be pumped quite airless, for which we used a Stuhl's mercury air-pump, which worked to our great satisfaction.

In this way there were finally obtained 3·5 litres gas, which then were condensed in liquid air. The condensing vessel which contained wires fused in, and the junction of the thermo-element, serving as thermometer, had the annexed form (see Fig. 2), and was pumped completely air empty before the gas was let in. The gas condensed itself very easy and complete under somewhat raised pressure (which is obtained by the raising of the mercury reservoir which is connected with the gasometer) to a transparent colourless fluid, in which floated small numbers of colourless crystals.*

Now the liquefied gas was raised to boiling-point by raising the condensing vessel out of the liquid air, and was fractionated into various gasometers, according to the boiling temperatures.

The temperature rose quickly from −189° to −181·2°; there it became tolerably constant, and about 2·5 litres of gas were here collected in bottles 2, 3, and 4, whilst bottle 1 contained that boiling between −189° and −181°. Then the temperature rose quickly to −153°, where the last portions of fluid evaporated. This part was collected in bottle 5. Finally, there remained a crystalline residue, which melted at about −147°,† and then quickly evaporated, and was collected in bottle 6.

Fig. 1.

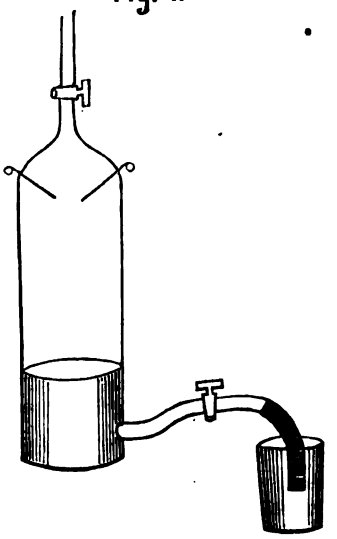
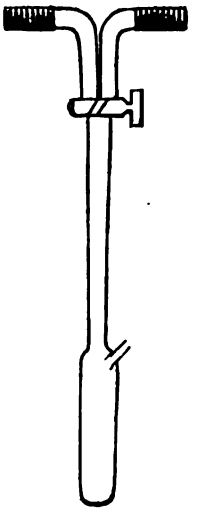


Fig. 2.



The gases from bottles 2, 5, and 6 were now examined with regard to their density, and also as to their spectrum characters.

Gas from bottle 2 (boiling-point −181·2°). This showed a very complete argon spectrum, in addition there was plainly visible the green krypton line, but not the yellow. The density of the gas was next found to be 38·6. But after the gas was mixed again with O, and sparked over soda-lye, then freed from O and dried, it showed a density of 40·34.

Gas out of bottle 5 (boiling-point −170° to 153°). Here also there was a beautiful argon spectrum, but on it was seen a very distinct krypton spectrum, in which the line D_4 and the green line were brilliant. The determination of its density gave 42·21 (O=32), from which it follows that we have to deal with a mixture of argon and krypton, in which the first forms the principal part.

The examination of the gas out of bottle 6 furnished unexpected results; here we saw a strong krypton

* Communicated by Hrn. van 't Hoff to the Berlin Academy of Sciences.

† See *Zeitschrift für Physikalische Chemie*, xxviii., 247. These are especially suitable for such purposes.

* Argon also condenses easily in liquid air at about 1½ atmospheres. The boiling-point at normal pressure (745 m.m.) is between −181·2° and −178·6° (compare against this, Olszewski, *Zeit. fur Phys. Chem.*, xvi., 383)). The inconstant boiling-point corresponds to the complete nature of argon.

† This melting-point determination can only be looked on as a provisional one, as the mass of the body was not sufficient to carry out an exact determination.

spectrum in which the lines D_4, 586·9 $\mu\mu$, and the green line 558·1 $\mu\mu$ were brilliant, like hydrogen lines. Besides, there were several very faint lines, which we were inclined to consider as argon lines. The question whether argon was really present appeared to us of importance, and its affirmation was so much more doubtful, as the characteristic argon line, according to our estimation, the orange line 603·3 $\mu\mu$, as also the most refrangible violet lines, were totally absent. We have therefore submitted our krypton spectrum to an accurate examination, and have measured a large number of lines, using a spectrometer which admitted of a reading to half a minute. We add here in column I. the red argon spectrum measured by Crookes; in column II. the argon spectrum measured by us; in column III. our krypton spectrum, and, finally, in column IV. the krypton spectrum mentioned by Ramsay and Travers, concerning which we may remark that in the latter are left out the argon lines observed at the same time (compare *Zeit. fur Phys. Chem.*, xxvi., 363).

It is seen on comparison that ten lines of our krypton spectrum, of which seven are in the red, are identical with argon lines, but that several of the fainter argon lines, together with the whole violet end of the argon spectrum, do not occur in our spectrum. As it appears to us, one must conclude therefrom that either very little argon or only a constituent part of argon is contained in our krypton.

	Argon. I.	Argon. II.	Krypton. III.	Krypton. IV.
Red.	764·6	764·4		
			761·5	
	750·6	749		
	737·7	738·3		
	726·3	726·7		
	—	716·3		
	—	715·3		
	705·64	708·8	707·6	
	696·56	697·3	697·3	
	684·2	688·9		
	675·4	676		
	666·4	669·2		
			645·2	
Orange.	640·7	642·3		
	637·7	638·3	638·1	
	630·2	631·9	631	
	628·1	—		
	621·0	623·2	624·3	
	617·3	617·1	617·8	
	614·3	614·6		
	609·9	610·6		
			608·3	
	605·6	605·7	605·7	
	604·5	—		
	603·8	603·3		
			601·45	601·1
			599·2	—
Yellow.	593·5	—		
	592·6	—		
	590·9	590·7		
	588·7			
			586·9	586·77
	585·8	—		
	583·4	—		
	580·3	—	—	582·9
	577·1	—		
	574·6	574·8		
Yellow-green.	568·3			
	565·1	565·5	565·9	—
	561·0	561·3		
	556·7	557·6	558·1	556·8
	555·7	556·8	557·3	556·0
	552·0	—		
	550·1	550·2		

	Argon. I.	Argon. II.	Krypton. III.	Krypton. IV.
Green.	549·65	—		
	545 6	546		
	544·4	—		
	542·1	542·5		
	525·8	525·6		
	522·2	522·0		
	518·58	519·2		
	516·5	516·5		
	506·5	—		
	501·2	—		
Green-blue.	496·55	—		
	493·8	—		
			492	
	487·9	—	491·3	490·9
			484·0	483·4
	—		482·5	483·0
		481·5		
Blue.			480·5	480·7
			473·6	473·6
	470·12	470·6	470·0	—
			467·5	467·1
	462·95	463·3	462·3	—
	459·45	459 7		
	451·4	—		
	450·95	451·6	450·5	—
Violet.			446·6	
			445·5	446·1
			437·5	438·7
			436·4	—
	434·5			
	433·35	433·7	432·5	431·7
	430·05	430·2		
	427·2	427·5	427·8	—
	426·6	426·8		
	425·95			
	425·15			
	420·10	420·5		
	419·8			
	419·15	419·2		
	418·3	418·4		
	416·45	—		
	415·95	416		
	415·65			
	404·4			
	394·85			
	390·45			
	383·55			
	377·15			
	363·25			
	362·37			
	362·28			
	360·50			
	356·65			
	356·28			
	355·45			

The density determination of our gas gave the figure 58·81 relative to oxygen 32. The observed data were the following:—

Weight of the gas, 0·0391.
Volume of the globe, 16·364 c.c.
Reduced barometric pressure, 739·4 m.m
Temperature, 19° C.

In view of the small quantity of gas and that the accuracy of our balance does not exceed 0·0001 grm., we estimate the error of our determination at 1 per cent.

For the control of this density determination, the result of which very much surprised us, we mixed the gas anew

with oxygen, and sparked over soda-lye. Hereby occurred a not inconsiderable volume reduction, so that we believed that our gas still contained nitrogen. But later it came out that our oxygen, which we considered nitrogen free, was not quite pure.

But we must here expressly point out that we now sparked so long that even after some hours no decrease of volume occurred, so that we could be sure that we had no more nitrogen in our gas.

Then it was freed from oxygen with pyrogallol and soda, and collected over mercury, where it was left standing over yellow phosphorus and fresh fused soda for forty hours. After this a new determination of density was carried out. The data were now the following:—

Weight of the gas, 0·0394.
Volume of the same, 16·364 c.c.
Temperature, 17·5°.
Barometer reduced, 743 c.m.

Therefrom the density comes out (referring to O=32), at 58·67, in remarkable accord with the first found figure, 58·81.

Therefrom it follows with certainty that the krypton contained no more oxygen, and since very little argon was mixed with it, as already inferred from the spectrum examination, one may well consider it as nearly pure. This conclusion is essentially supported by the fact that this gas originated from a crystalline body, which doubtless speaks for its not being a mixture.

Then comes the question, what position does krypton occupy in the periodic scale if we consider the density 58·8 as nearly correct? We believe that we should place the new group of elements in air before group I., so that He=4 before lithium, Ne=20 before sodium, A=39 before potassium, and, finally, Kr=59 before copper.

Still this is only a hypothesis which requires further testing. Herewith we are busy, as perhaps through the solid condition of krypton there offers a newer and much more simple road for its preparation, which we have already entered upon.

This consists in the working up of the well separated constituents of the liquid air which evidently will contain krypton next to carbon dioxide, when the solubility in liquid air does not suffice to hold in solution all the krypton present.

OBSERVATIONS ON THE EFFECT OF DESICCATION OF ALBUMIN UPON ITS COAGULABILITY.*

By J. BRETLAND FARMER, M.A., Royal College of Science, London.

It has been known for some time that it is possible, under certain circumstances, to expose seeds to the influence of high temperatures without thereby necessarily destroying their power to germinate. Some experiments in this direction were conducted at the Royal Gardens, Kew, some years ago by Dr. Morris, but the results, although of much interest, do not appear to have been published. However, the seeds were exposed to the action of boiling water, and even to a higher temperature in an oven, without losing their ability to germinate when the ordeal was over.

It has been noticed, in heating seeds in water, that if the seed-coat through any cause becomes ruptured, or if it softens and swells, the seeds which are thus affected are incapable of manifesting any further evidence of vitality. It appears to me that a fair inference to be drawn from these facts is that the admission of water to the living cells is a potent factor in bringing about their death.

Jodin (*Comptes Rendus*, 1899) has recently communicated some facts which point to the same conclusion. He exposed seeds of pea and cress to a temperature of 98° C., and found that, unless great care had been previously exercised to ensure the dryness of the seeds, they were all killed. When they had been previously dried he succeeded in subsequently germinating 30 per cent of the peas and 60 per cent of the cress seeds. Perhaps the disproportion in favour of the latter may, at least in part, be ascribed to their small size, and consequently to the less difficulty in sufficiently drying the seeds.

It would seem to follow, from what has been said, that the instability of the complex molecular structure of which living organisms are made up may be lessened by appropriate desiccation, but the substances concerned are too complex to render themselves readily accessible to inquiry. It appeared, however, that it might be worth while to study the effects of desiccation on albumin from this point of view. Albumin is not only a highly complex proteid, and perhaps in some respects akin to protoplasm itself, but it is one which gives tolerably definite heat reactions. It is in connection with the last-mentioned point that the new facts in this paper are specially concerned.

It is of course known that albumin in a watery solution is readily coagulated on heating to a certain temperature. This temperature, however, is not necessarily constant for even one type of albumin, doubtless owing to the readiness with which it undergoes change. Thus albumin obtained from different hens' eggs will often be found to coagulate at different temperatures, and the differences appear—in part, at any rate—to be connected with the age of the egg. I have found, in the case of freshly-laid eggs, that the characteristic opalescence which marks the early stages of coagulation may set in as low as 60° C., the clotted coagulum being fully formed at 64° C. The heat was applied by means of a large water-bath, so as to ensure its being as uniform as possible. Another sample of albumin from a different egg, tried simultaneously and under the same conditions, only exhibited opalescence at 65·6° C., and coagulated completely at 68° C.

The albumin on which most of my experiments were made was obtained from Merck, of Darmstadt, and was sent as dried egg-albumin. It readily dissolved in water, with the exception of a little flaky insoluble portion, which was filtered off. The solution had a low coagulation-point, the opalescence appearing at 60° C., and the clot at 62° C. to 63° C.* Filtering off the clot and testing the filtrate at higher temperatures yielded no further coagulation.

If a sample of this (dry) albumin be placed in a flask, the mouth of which is furnished with a cork and attached to a set of drying tubes, and the temperature of the flask raised to 80° C., a short exposure, of at any rate two to three hours, is enough to completely alter the albumin. The object of the drying tubes is to prevent any more moisture than is already present in its substance reaching the albumin from the steam or from any other source. Thus heated, the albumin is found to have become insoluble in water, and in fact to have undergone a change corresponding to coagulation.

If, however, the albumin be carefully *dried* before being subjected to these conditions, the results are quite different. For the present purpose it was found to be sufficient to expose a thin layer of albumin in a glass dish to a temperature of 52° to 55° C. in an incubator. This ensures a very thorough desiccation. The process may be hastened by introducing a vessel of sulphuric acid, though this precaution was not found to be necessary. Thus dried, the albumen loses its shellac or glue-like appearance, and easily crumbles to very small particles.

On comparing the solubility and coagulability of this

* A Paper read before the Royal Society, April 5, 1900.

* It is of course known that several factors affect the coagulation-point. The figures given represent those obtained in my experiments, which were all kept as uniform as possible so as to eliminate the factor of variability.

specially dried material with the ordinary sample, no difference could be detected in any respect.

Numerous experiments were made with this dried material, of which the following may be taken as typical. It may be added that the results throughout were almost surprisingly uniform in the different experiments made.

A sample of the specially dried albumin was introduced in a flask so as to form a thin layer over the bottom. The flask was connected with drying tubes filled with calcium chloride, and with phosphorus pentoxide. The flask was warmed and cooled rapidly several times, in order to cause the contained air to circulate through the drying tubes.

The temperature of the flask was then raised in a brine-bath to 102° C., and kept at this temperature during the whole of one day (six hours). Next day, and without opening or disturbing the apparatus, the temperature was again raised to 107° C., and finally to 110° C. It was maintained between these limits for seven hours; thus the contents of the flask had been for thirteen hours exposed to a temperature of considerably over 100° C.

On testing the albumin it was found to be soluble in water, and in no way, as far as could be observed, did it differ from the unheated material. On gradually warming the solution side by side with a similar solution of the unheated albumin, both became opalescent at a temperature of 60° C., and both were completely coagulated at 62° C.

It thus appears that, if precautions are taken to ensure appropriate desiccation, it is possible to heat albumin for, at any rate, thirteen hours to a temperature varying between 102° to 110° C. without producing any obvious change in its ultimate molecular (or micellar ?) structure. It made no difference to the result whether the heat was gradually or rapidly applied. Thus, in one experiment, the temperature was raised from 50° C. to 103° C. in fifteen minutes, and in other examples the flask was withdrawn from the hot bath, cooled, and suddenly re-immersed. How much higher the temperature could be raised without producing an obvious effect, I am not prepared to say; nor did I investigate the action (if any) which might possibly be produced by a much longer exposure to heat within the limits already mentioned. This formed no part of my object, which was primarily to try to get a point of comparison between the complex seed and the simpler but still very complex proteid.

Other experiments were made in order to test the sensitiveness of the albumin to small quantities of moisture.

For this purpose, two flasks attached to drying tubes were used, one of them serving as a control experiment, and remaining unopened until the end. The other was opened three times, and a small sample taken out each time. By this means the ordinary air of the room obtained complete access to the albumin. The duration of the experiment was ten hours. The first sample was withdrawn after the flasks had been heated to 102° C. for three hours; it dissolved and coagulated normally. A second sample was withdrawn after three hours more, and it was found that whilst it dissolved and became opalescent on heating to 60° C., the coagulation change did not at once set in, but the opalescent solution became more milky and of a deeper fog-yellow by transmitted light, finally coagulating at about 68° C. A third sample taken out at the close of the experiment (*i.e.*, four hours after the last opening of the flask) also dissolved, became slightly opalescent at about 64° C., but did not coagulate even at 90° C., although the opalescent milkiness became very pronounced. Viewed by transmitted light, the solution was translucently yellow. Even boiling failed to produce anything which could be fairly termed a coagulum. It appeared probable that the admission of watery vapour had permitted the inception of the changes which normally, at high temperatures, result in coagulation; but in this case they were arrested, some precursor of alkali-albumin being probably produced, as is often the case on slowly coagulating albumin solutions. Under these circumstances, however, the entire mass of the albumin had undergone this change. This supposition turned out to be correct, for the addition of a trace of acetic acid at once caused the solution to be susceptible to coagulation at about 60—62° C.* Hence it is fair to infer that although the slight amount of moisture introduced during the opening of the tube did not suffice to enable complete coagulation to occur, it did permit the early changes to begin, and to slowly, and in a modified way, to affect the entire mass. This experiment was repeated several times, and always with the same result.

It seems difficult, in the light of the foregoing observations, to resist the inference that in the complete absence of moisture albumin may be reduced to a state of relative molecular (or micellar) immobility; the re-arrangements which, in the presence of water, and at a sufficiently high temperature, normally take place in its ultimate structure being held in abeyance during the suspension of the essential condition of the presence of sufficient moisture. The substance is brought, so to speak, into a static condition; chemical or physico-chemical change is inhibited, just as is an interaction between phosphorus and oxygen when conditions of complete dryness obtain. It is tempting to extend these considerations to the case of seeds and spores, (*e.g.*, of certain bacteria), and to ask whether similar conclusions may not be fairly assumed to obtain there, for it may well be a fact that the protoplasm, like the albumin, which is at any rate akin to it, when sufficiently desiccated withstands conditions which otherwise would certainly promote chemical disintegration. They, too, appear to be reduced to a "static" condition by drying, and the researches of Romanes (*Proc. Roy. Soc.*, vol. lvii.), indicated no measurable chemical change as proceeding in them under these circumstances; and, again, the investigations of Brown and Escombe (*Proc. Roy. Soc.*, vol. lxii.), and of Sir W. Thiselton-Dyer (*Proc. Roy. Soc.*, vol. lxv.), have also rendered it difficult to believe, when subjected to the other end of the scale of temperature, that any metabolism can really be proceeding. In these cases the molecular machinery of life is all present and intact, but the *manifestation of vitality*, as measured by chemical movement, and by the change in the condition of energy, is absent. But such a state differs widely from death, seeing that when the conditions favourable to the continuous progress of those reactions which are associated with vitality are restored, the organism proceeds to work in the normal manner once more. Similarly the albumin heated in the desiccated form retains, instead of changing, that particular molecular condition which enables it, on restoring the essential conditions of moisture, to coagulate in a normal fashion when heated to a suitable degree of temperature.

ON THE

ESTIMATION OF THALLIUM AS THE ACID AND NEUTRAL SULPHATES.†

By PHILIP E. BROWNING.

Crookes (Chem. News, viii., 243) has shown that the salt obtained by heating thallous chloride with sulphuric acid until the excess of the latter is expelled, and then raising the heat to redness, has the constitution of a neutral sulphate.

He also found that continued heating did not result in any essential loss of weight, and suggested the possibility of applying this method of treatment to the estimation of thallium.

Castanjen (*Amer. Journ. of Science*, viii., 460), in a

* A solution of albumen treated with a very small quantity of a dilute solution of potash undergoes a similar change. The substance formed is not true alkali-albumen, since no precipitate is produced on neutralising, and a coagulum on heating this neutralised solution.

† Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, February, 1900.

recent paper, discusses thoroughly the compounds of thallium, and confirms essentially the statements of Crookes in regard to the neutral sulphate, adding, however, the observation that on strong ignition in the air this salt tends to lose sulphuric acid. He also mentions in the same paper the acid sulphate, and states that on heating it first melts, and on continued heating gives off sulphuric acid, leaving the neutral sulphate.

The work to be described in this paper was undertaken to determine under what conditions the formation of these salts may be applied to the estimation of thallium. For the work a solution was made by dissolving a given amount of the nitrate in water and making up to a litre. The value of the solution was determined by precipitating measured and weighed amounts of this solution both as the iodide and chromate, as described in a previous paper (*Journ. f. Prakt. Chem*, cii., 131). Closely agreeing results by both methods were taken as the standard. Measured amounts of this solution were drawn from a burette into weighed platinum crucibles, and the weight taken as a check on the burette reading. To the solution in the crucible a few drops of sulphuric acid was added, and the water removed by evaporation over a steam-bath. The crucible was then removed to a radiator, consisting in this work of a conical iron cup, and heated at a temperature ranging from 220° C. to 240° C., until fuming ceased and the weight after half-hour periods of heating remained constant. The crucibles were placed in a radiator upon a pipe-stem triangle so that they were about 5 c.m. from the bottom, which was heated at low redness. A thermometer hung so that the bulb occupied the same position as the crucible gave the reading mentioned above.

As will be seen, the results obtained by this treatment agree closely with the calculated amounts of acid sulphate which should be formed. In several experiments this salt was dissolved in water, and the sulphuric acid present in combination precipitated by barium nitrate. The results obtained agreed closely with the formula of the acid sulphate of thallium. Having obtained by the method described the acid sulphate, the crucibles were removed and heated carefully over a free flame to low redness, when, after a considerable evolution of sulphuric acid fumes, the weight again became constant, and the results showed a condition closely approximating to that of the neutral sulphate. In several of these experiments the sulphuric acid present in combination was determined and showed amounts closely agreeing with the constitution of the neutral sulphate.

	$TlHSO_4$ Calculated.	$TlHSO_4$ found.	Error.	Tl_2SO_4 calculated.	Tl_2SO_4 found.	Error.
	Grm.	Grm.	Grm.	Grm.	Grm.	Grm.
1.	0·1605	0·1596	0·0009 −	0·1344	0·1346	0·0002 +
2.	0·1611	0·1608	0·0003 −	0·1349	0·1346	0·0003 −
3.	0·1608	0·1608	0·0000 ±	0·1347	0·1352	0·0005 +
4.	0·1612	0·1600	0·0012 −	0·1350	0·1346	0·0004 −
5.	0·1602	0·1596	0·0006 −	0·1341	0·1346	0·0005 +
6.	0·1608	0·1596	0·0012 −	—	—	—
7.	0·1617	0·1604	0·0013 −	—	—	—
8.	0·1608	0·1592	0·0016 −	0·1347	0·1358	0·0011 +
9.	0·1609	0·1590	0·0019 −	0·1348	0·1346	0·0002 −

These results would seem to show that thallium may be estimated either as the acid sulphate or as the neutral sulphate by careful attention to the proper conditions of temperature.

The Hydrates of Chloride of Manganese.—P. Kouzenezoff.—The author describes a new hydrate, $MnCl_2.6H_2O$, which is obtained by cooling a solution containing, $MnCl_2 + 11{\cdot}7H_2O$ down to −21°. By shaking violently we notice the appearance of a more or less distinct cloud which slowly changes into almost colourless crystals, sometimes reaching a length of 5 m.m. This hydrate decomposes at −2°.—*Journ. Soc. Phys. Chim. R.*, xxx., 741.

THE DETERMINATION OF NICKEL IN NICKEL-STEEL.

By GEO. WM. SARGENT.

The results obtained in this laboratory by the following method have been such as to warrant my placing it before fellow chemists, who, I believe, will find the method more reliable and more pleasant to work than most procedures now in vogue for the determination of nickel in steel.

The method in detail is as follows:—Dissolve 2 grms. of the steel in hydrochloric acid (1·1 sp. gr.), add 1 c.c. of strong nitric acid to oxidise the iron, and evaporate to hard dryness. Take up the residue in 20 c.c. of hydrochloric acid (1·1 sp. gr.), evaporate to 10 c.c. volume, and transfer the solution to a 250 c.c. separatory funnel (see method of Chase for the "Determination of Nickel and Aluminium in Steel" in the Appendix of "The Chemical Analysis of Iron," by Blair, Third Edition). Use warm hydrochloric acid (1·1 sp. gr.) for washing the contents of the beaker into the funnel, taking care to keep the volume as small as possible. Cool the funnel and its contents under the spigot, then introduce 40 c.c. of ether free from alcohol, which has just been thoroughly shaken with 5 c.c. of strong hydrochloric acid, and shake vigorously for ten minutes, keeping the temperature from rising by frequently holding the funnel under the running water. Allow the funnel to stand a few minutes, draw off the lower aqueous layer containing the nickel, copper, manganese, and other chlorides not held by the ether, wash down the sides of the separatory funnel with hydrochloric acid (1·1 sp. gr.), shake with the ether, and run the washings into the beaker with the chlorides of nickel, copper, &c. Two washings with 5 to 10 c.c. of hydrochloric acid are sufficient to completely remove the nickel and other chlorides from the funnel. The ethereal solution containing the ferric chloride is run into a residue bottle, and later the ether recovered by distillation. Boil the liquid containing the nickel to completely expel the ether and add to the boiling solution, diluted to at least 200 c.c., an excess of ammonia and 10 to 20 drops of bromine water, to precipitate any iron or manganese. Filter, wash, re-dissolve in hydrochloric acid, re-precipitate with ammonia and bromine-water, and filter. Combine the filtrates, boil, remove any precipitate, acidulate with hydrochloric acid, boil until the excess of bromine has been expelled, and precipitate the copper as sulphide. The filtrate from the copper sulphide, which contains nickel only, is evaporated to a volume of 100 c.c., cooled, and 1 c.c. excess of ammonia added. Now introduce into the nickel solution, which is best contained in an Erlenmeyer flask, 5 c.c. of silver nitrate ($\frac{1}{2}$ grm. silver nitrate in a litre of water), and the same amount of a 2 per cent solution of potassium iodide. Run into the opalescent solution, which should have a temperature slightly lower than that of the hand, standard potassium cyanide (1 c.c. equal to about 0·001 grm. of nickel) until the liquid becomes clear and bright. This titration is best made with a black background, when the end-reaction becomes very sharp and decided after a little practice, half a drop only being sufficient to discharge the opalescence (see "Determination of Nickel in Nickel-steel," by E. D. Campbell and W. H. Andrews, *Journ. Am. Chem. Soc.*, xvii., 127).

The potassium cyanide solution is standardised by introducing into an Erlenmeyer flask 10 c.c. of a solution containing a known amount of re-crystallised nickel nitrate, $Ni(NO_3)_2.6H_2O$, 10 c.c. of hydrochloric acid (1·1 sp. gr.) 1 c.c. excess of ammonia, 5 c.c. each of the silver nitrate and potassium iodide, diluting the whole to 100 c.c., and running in the potassium cyanide until the solution clears. It is necessary that a blank be made upon the silver nitrate and potassium iodide used as the indicator, under the same conditions as the standardisation, and this amount deducted from each titration.

The presence of varying quantities of ammonium chloride in the nickel solution to be titrated has no effect on the titration. This is evident from the following:—

Taken.			Found.				
NH_4Cl present.	$Ni(NO_3)_2$.	Weight of Ni.	Readings. First.	Readings. Second.	Difference.	Blank.	Weight of Ni.
Grms.	C.c.	Grm.					Grm.
1	20	0·02	0·0	20·2	20·2	0·45	0·01996
3	20	0·02	20·2	40·5	20·3	0·45	0·02007
7	20	0·02	0·0	20·2	20·2	0·45	0·01996

1 c.c. potassium cyanide solution = 0·001011 grm. nickel.

Some of the results obtained by the foregoing method I append:—

Mark.	Solution of KCN equivalent to Ni.	Weight of nickel found.	Nickel.
	C.c.	Grm.	Per cent.
389	27·65	0·02795	1·397
389	27·65	0·02795	1·397
389	27·55	0·02785	1·392
390	28·60	0·02891	1·445
390	28·55	0·02886	1·443
382	30·40	0·03073	1·536
382	30·20	0·03053	1·527
387	31·00	0·03134	1·567
387	30·70	0·03104	1·552
387	31·00	0·03134	1·567
109	51·00	0·05156	2·578
109	51·30	0·05190	2·595

1 c.c. potassium cyanide = 0·001011 grm. nickel.

The above determinations were made on steels in which the manganese did not exceed one-tenth per cent, and in which there was no copper.

Different amounts of nickel were mixed with solutions containing 2 grms. of iron and the nickel determined therein:—

Taken.		Found.	
$Ni(NO_3)_2$ solution.	Weight of nickel.	KCN solution equivalent to Ni.	Weight of nickel.
C.c.	Grm.	C.c.	Grm.
30	0·03	29·60	0·02992
30	0·03	29·05	0·02937
40	0·04	39·60	0·04003
40	0·04	39·70	0·0401
45	0·045	44·05	0·0445

For the estimation of nickel in ferro-nickel, dissolve 20 grms. of the sample in aqua regia, evaporate to hard dryness, take up with hydrochloric acid (1·1 sp. gr.), transfer to a litre flask, and dilute to the mark with water. Remove 50 c.c., to which add 5 c.c. strong hydrochloric acid, and concentrate to one-sixth its volume. Transfer to the separatory funnel, and proceed as with the steel.

A sample of ferro-nickel which, by an unknown method, contained 33·93 per cent of nickel, yielded 33·80, 34·00, 33·88, and 33·96 per cent. Another sample which, according to one chemist, contained 34·95 per cent of nickel, gave 35·15 and 35·26 per cent.

The above-described method is, as can readily be seen, a combination of a modified portion of the method of Chase, with a slightly changed part of the procedure given by Campbell and Andrews. In the course pursued by Chase, two ether separations are necessary to remove the ferric chloride, while the separation, as worked by us, need not be repeated; but little ferric chloride is left, and the two ammonia precipitations are sufficient to completely remove the iron from the nickel.

With regard to the titration with potassium cyanide, that may be accomplished in the presence of most all the salts of ammonium and the mixed alkalis except the nitrates. The excess of ammonia should not in any case exceed 3 c.c. (see "Estimation of Cyanogen," by Wm. J. Sharwood, *Journ. Am. Chem. Soc.*, xix., 415).

While in steels about which one knows nothing, it will be necessary to hunt for copper, yet, in the general run of furnace work, copper is absent, and knowing such to be the case, the working with the unpleasant gas, hydrogen sulphide, is done away with entirely. As far as time is concerned, the method is short, requiring no longer time than the method of Chase.

In conclusion, I wish to here acknowledge the assistance of Mr. Clarence Ebaugh and Mr. John K. Faust in working out this method. — *Journal of the American Chemical Society*, xxi., No. 10.

Laboratory of the Carpenter Steel Co.,
Reading, Pa.

A NEW PHOSPHATIC MANURE; PREPARED PHOSPHATE OF ALUMINA.

By M. ZECCHINI.

Some months ago a new phosphatic compound was placed on the market under the name of *prepared phosphate of alumina*, to which was attributed an efficacy equal to that of Thomas's scoria; in fact, this new material was intended to replace the latter, and at a price, per unit of phosphoric acid, equal if not superior to that of scoria.

My advice having been sought by several companies and syndicates, with regard to this manure, I decided to carry out a few comparative culture experiments so as to learn its efficacy, and at the same time I examined its composition, as well as its behaviour under the action of the reagents most commonly used nowadays for determining the assimilability and efficacy of the phosphatic compounds as fertilising materials.

I ought to add that, in spite of my repeated enquiries with regard to the origin and source of this substance, I have not been able to obtain any information whatever on this subject. I am therefore quite ignorant as to whether it is simply a pulverised mineral or a manufacturing residue, as the word "prepared" might lead one to believe.

The material, as it exists commercially, is in a fine powder of a dirty rose-colour, with an amorphous appearance. Analysis has given me the following results:—

Water lost, between 100° and 110°	7·21 p. c.
Do. at red heat	0·12 ,,
Residue insoluble in HCl	12·63 ,,
Phosphoric anhydride	42·22 ,,
Alumina	25·45 ,,
Ferric oxide	11·90 ,,
Lime	0·35 ,,
	99·88 ,,

The part insoluble in hydrochloric acid has the following composition, in proportion to 100 parts of the original material:—

Silica	10·32 p. c.
Oxides of iron and aluminium	1·38 ,,
Lime	0·55 ,,
Magnesia	0·32 ,,
	12·57 ,,

This composition, expressed in parts per 100 of the insoluble substance, gives the following figures:—

Silica	81·71 p. c.
Oxides of iron and aluminium	10·92 ,,
Lime	4·35 ,,
Magnesia	2·53 ,,
	99·51 ,,

I ought to mention that a few qualitative analyses have shown me that I had to deal with an orthophosphate, and, further, that the material was easily soluble in hot soda or potash lye, leaving only an insoluble residue of silica and hydrate of iron.

From these analytical results we gather the following facts:—That the phosphate under examination should be considered as a neutral or basic phosphate of iron and alumina; and, secondly, that we have probably to do with a natural phosphate which has simply been ground.

There can be no doubt, however, that it cannot be looked upon as a superphosphate or a bibasic phosphate. In the first case it would contain at least 1 part of phosphoric anhydride soluble in water at the same time as the sulphates, which is not the case; but at any rate it ought to contain a large quantity of phosphoric anhydride with regard to the proportion of bases present.

The amount I found is quite insufficient to correspond with the oxides of iron and aluminium, if we wish to consider these two bodies as the neutral phosphates $Fe_2P_2O_8$ and $Al_2P_2O_8$ respectively. The quantity to effect this would be at least 45·99 per cent, while only 42·22 per cent of phosphoric anhydride was found.

It is true that a certain portion of the oxides of iron and aluminium found, might result from the partial attack of the silicate by hydrochloric acid, but the proportion would not be so great considering the amount of silica present; consequently it is very probable, as I have already stated, that we have to deal with a neutral or basic orthophosphate of alumina in which this base is partially replaced by ferric oxide.

Further, I ought to mention that the phosphates of alumina, or of alumina and iron, known as minerals,—such as wavellite, redondite, &c.,—which are, as a matter of fact, basic phosphates of alumina and iron, have the property, as has also the substance under examination, of dissolving easily in the alkaline lyes. I am therefore of opinion that it is very probable that the material I have examined should be classed as a natural phosphate analogous to those I have just mentioned.

But putting on one side this purely inductive reasoning, we come to the manner in which this material behaves with reagents which show the assimilability of phosphoric acid. With this object in view I tried the citric solutions which are now commonly used for scoria—that is, Wagner's acid citrate of ammonia, and citric acid at 2 per cent. These two reagents were specially prepared with the necessary precautions, and were consequently perfectly fresh.

In each case I placed in a half-litre flask 5 grms. of the substance, and then added the solvent up to the mark, gently shaking to prevent it becoming stuck to the bottom, after which I subjected the flask to an hour's rotation in the well-known apparatus used for this purpose.

The quantities of phosphoric anhydride dissolved were absolutely minimal,—that is to say, 0·32 per cent with Wagner's citrate, and 1·34 per cent with 2 per cent citric acid, as means of a number of very concordant experiments.

I must not, however, pass in silence over a very curious occurrence I observed with regard to the behaviour of this phosphate with the solution of acid citrate of ammonia by Wagner's method.

Having, as I have said, made an estimation in the ordinary manner with perfectly fresh solutions properly prepared, the solubility was practically *nil*, both when using a concentrated solution (5 grms. of material to 500 c.c.) and a dilute solution (200 c.c. of solution plus 300 c.c. of water to 5 grms. of substance), as is done with scoria.

Having, then, once by accident used some acid citrate of ammonia, which had been left for some months in a flask, and which showed some mouldy growths (*penicillium*), the solubility of the phosphate increased enormously, the proportion of the substance dissolved being 25·34 per cent, although I had used the solution diluted in the proportion of 2 to 5, as is done with scoria.

I decided to repeat the experiment, so left some acid citrate, again for some months, in such a manner as to encourage the growth of moulds. I repeated the experiment under the same conditions, and obtained a still greater quantity of the substance in solution,—that is to say, 29·95 per cent.

The fact is very curious, as the solutions remained acid; not having, however, examined the reaction any further, I am unable to offer any definite explanation. It may be possible that, by the action of the moulds, other salts of ammonia are formed having a stronger dissolving effect on the phosphate than the citrate of ammonia. I intend following up this matter.

I then conceived the idea of trying neutral citrate, such as is prepared at our agronomic stations for the examination of superphosphates. In this experiment I acted on 5 grms. of material with 100 grms. of the reagent, diluted with 150 c.c. of water, always in flasks of 250 c.c. capacity, which I submitted to rotation for an hour in the usual manner. In this way I obtained 0·86 per cent of dissolved phosphoric anhydride.

Having obtained this result in the cold, I now tried the effect of heat. I took 5 grms. of material and placed them in a flask of 250 c.c. capacity, with 100 c.c. of neutral citrate, making up the volume with distilled water. The whole was kept on the water-bath at 40 to 45° for one hour, with frequent agitation. Under these conditions 5·47 per cent of phosphoric anhydride was dissolved. As can be seen, even with neutral citrate, the solubility is low.

I then tried the use of ammoniacal citrate of ammonia, prepared according to Petermann, by reacting for an hour in the rotatory apparatus with 250 grms. of the reagent on 2·5 grms. of material. In this case the amount of phosphoric anhydride dissolved was considerable, and reached 25·79 per cent.

This fact led me to think that the solubility of the phosphate of iron and alumina depended on the alkalinity of the solution, and to decide this point I tried the following experiments:—

Into four 250 c.c. flasks I placed respectively 5 grms. of the material and 100 c.c. of standard neutral citrate. In the first I made up the volume with distilled water, while, before doing this to the other flasks, I added respectively 5, 10, and 20 c.c. of ammonia of 0·92 density. All the flasks were rotated for an hour, and after filtration I precipitated the phosphoric acid by means of magnesia mixture in all the solutions.

In this manner I obtained the following quantities of dissolved phosphoric anhydride:—

Neutral citrate alone	0·86 p. c.
Do. + 5 c.c. NH_3	18·85 „
Do. +10 „ „	31·17 „
Do. +20 „ „	31·23 „

It is thus evident that, whether hot or cold, to make any considerable proportion of this phosphate soluble, it does not suffice to use concentrated citrate of ammonia, but it is necessary for this salt to have a strongly alkaline reaction.

Taking count of this, I endeavoured to find whether the solubility of the phosphate could be still further increased by diminishing the proportion of the substance with regard to the quantity of reagent.

I therefore made two experiments in which, while maintaining the conditions of the preceding experiment,—that is to say, with 100 c.c. of standard neutral citrate in flasks of 250 c.c. capacity, and adding 20 c.c. of ammonia,—I only used 2·5 grms. of material instead of 5 grms. as before. In one of the experiments the flask was kept cold and rotated for an hour. In the other experiment I heated the flask to 40 to 45°, with frequent agitation for an hour. In the first case I obtained 31·93 per cent, and in the second case 36·67 per cent of soluble phosphoric acid.

This shows that heat aids the solution of the phosphate. In my opinion the above is the process which should be adopted in the examination of this substance, the more so as I have proved that the ordinary natural phosphates (phosphorite, apatite, &c.), reduced to fine

powder and treated in the same manner, give up to the solvent only traces of phosphoric anhydride. I will not dwell longer on this subject, nor deduce any too absolute conclusions from the practical point of view, as my firm conviction is that our reagents are badly chosen for the determination of the true fertilising value of a manure, and especially of these phosphates, as from this point of view the real proof can only be shown by experiments on culture.

In my opinion laboratory experiments should only be taken as showing the relative chemical differences in the nature of the materials examined.

It is also no less true that the behaviour of the phosphate of alumina is different to that of scoria, which is very soluble in acid citrate, in solutions of citric acid, and (as I have already had occasion to show) even in neutral citrate, while the phosphate of alumina may be looked upon as insoluble in these reagents.

Thus, the solubility in a distinctly alkaline citrate is so far removed from that which may occur in nature, by means of osmotic phenomena with the acid juices of the roots of plants, that it cannot be considered as affording much proof with regard to the fertilising value of the substance.

It should be further noted that this phosphate contains hardly any lime, even combined, and none at all free: this, without doubt, is an important coefficient of the efficacy of scoria. For this, as well as for other reasons, it seems to me that it would at present be at least premature to make an absolute comparison of this *prepared phosphate of alumina* with scoria. But this is not a sufficient reason to refuse it, *à priori*, the character of having a distinct fertilising power. It is in a very fine state of division; and in such a condition even the natural phosphates of lime, although insoluble in water and only slightly soluble in the ordinary citric solvents, and neutral or alkaline citrates, give good results in practice, especially when well mixed with the soil by thorough digging.

A sound opinion, thus, cannot be given with regard to this new phosphatic manure, except after systematic practical experiments. I have, as I have said, started such experiments, and hope soon to be able to describe the results. It seems to me, however, that it would for the present be more prudent to give this phosphate of alumina a value intermediate between that of scoria and that of the natural phosphates.—*La Chimica Industriale*, Jan., 1900.

THE CHEMICAL ACTION OF LIQUID SULPHUROUS ACID ON IRON.

By A. LANGE.

I HAVE already dealt with this subject in the *Zeitschrift für Angewandte Chemie*, and at the end of the article I emphasized the fact that the attacking of the iron by sulphurous acid would be particularly objectionable in the case of ice-making machines, and the necessity of keeping the acid absolutely anhydrous. As far as I am aware no importance has been attached, up to the present, to the proportion of water in the sulphurous acid used, either because the objections to it were not known, or because, in face of the present state of our knowledge, it was not easy to admit that sulphurous acid was capable of dissolving water. Now, water gets into the machine while at work, the more easily when there may be accidental falls of pressure in the compressor, exactly as has been shown with machines using ammonia, but no general explanation, covering all cases, has been given. But the moment the sulphurous acid circulating in the machine contains water, the attack of the iron should take place as I have observed, and this is helped by the high temperature of the compressor, if the action is not prevented by other causes. I believe I have found one cause in the cooling generally applied to ice-making machines, using sulphurous acid, and especially in the neighbourhood of the cylinder and the piston. It must be admitted, and I have insisted on this point in the *Zeitschrift für die ges. Kälteindustrie* (May, 1899), that the sole object of the cooling is to keep these parts at a lower temperature than that at which the attack might take place with a somewhat impure sulphurous acid. It is always the cocks of the compressors, which cannot be protected in this manner, that should show traces of the attack by the action of aqueous sulphurous acid; still, as far as I am aware, such action has not been observed. Now, a little while ago, I received some material taken out of the compressor of a sulphurous acid machine, one which certainly had not been taken very much care of; I was asked, at the same time, if this substance would help me to explain how the corrosion took place. The material was black and friable, and lost 3 per cent of its weight in the desiccator and 14 per cent at 100°. By extraction it gave 15 per cent of an easily solidified fat. The solution obtained by treating the residue with cold water and filtering was neutralised with hydrochloric acid, and submitted to prolonged boiling. I thus obtained a precipitate coloured black by sulphide of copper. The solution thus contained copper. I easily obtained the reactions I have already described showing the presence of sulphurous and hyposulphurous acids and protoxide of iron. The substance further contained a large proportion of graphite, which is apparently due, like the fat extracted from the lubricating material, to the material used for making the cocks and pistons air-tight; a small proportion of it may also be due to the attack of the iron.

The presence of hyposulphite of protoxide of iron in the products of the corrosion establishes, in a decided manner, the fact that this latter is caused by the chemical action of the sulphurous acid on the iron, and that it is not the result of a mechanical action.

It was also evident, according to my experiments, that the attack took place owing to the presence of water in the acid; it was, however, important to determine this factor for the acid in the machine. The engineer having told me that water was frequently drawn off from the machine, I was led to admit that the acid was saturated with water, a saturation which my experiments showed to be 1 per cent.

However, one sample gave 0·2 per cent, and another taken some days later—0·45 per cent of water; now the sample taken in both cases were not very good ones, and these figures it should be admitted are rather too high than too low. As the samples were brought in open vessels from the machine, a part of the sulphurous acid would escape in vapour, and thus cool the remainder below its boiling-point at the atmospheric pressure. Although we have not, as in the case of ammonia, made comparative experiments on the variations which may take place under these conditions, we are bound to admit that it is the pure acid which escapes, and then we should find in the remaining acid a proportion of impurities rather too high than too low. The samples obtained were cooled considerably; under these conditions the first became cloudy, with a deposit of small oily drops; on the surface of the second, small crystals appeared, probably a hydrate of sulphurous acid. The cooled samples were transferred to pipettes with taps, which had previously been exhausted of air, so that there was nothing to fear with regard to variation in composition. I must here draw attention to a phenomenon which occurred during the course of these researches, as well as in my previous research, either with pure sulphurous acid or with the acid diluted with water, and which I have not yet mentioned. If we allow the sulphurous acid to run out of the pipette extremely slowly, no change is observed in the acid remaining liquid; but if we open the tap in such a manner that there is a rapid volatilisation, so that the outside becomes covered with dew, the liquid becomes

cloudy, and thus crystalline deposits form round the sides of the vessel: these cakes always form a little above the surface of the liquid, and gelatinous or crystalline fragments break off and fall into the liquid, and float therein. When once the whole of the acid is distilled, and the pipette become warm, nothing but oily drops remain round the sides. These crystallisations are probably due to dissolved fat, and to the formation of hydrates of sulphurous acid. Now, how does it happen that, under these circumstances, a relatively great degree of purity of the sulphurous acid was noticed at the time of taking the sample, while the machine certainly contained strongly aqueous acid, and that seeing the amount of the product of corrosion for a considerable time? We may give the following answer to this question, an answer which will also explain why the action I suspected and observed either did not take place or did so only by accident, if the machine was working properly. By the cooling of the hot compressed gas leaving the compressor, there condenses in the upper part of the refrigerator (condenser), at the same time as the warm liquid sulphurous acid, the water eventually found. Now, as the warm, liquid, sulphurous acid is not able to dissolve so much water as the liquid acid at 15°, and that, further, the specific weight of the acid is to that of the water as 3 to 2 (about), the water remains in the upper part. Besides this, the fresh quantities of acid coming from the compressor are cooled in the same manner, and the condenser—serving also as a reservoir—remains always filled up to a certain height; in this way the water always remains above the reserve of acid which gradually descends, and it is only after having been thus purified that it is again cooled in the condenser without having had the opportunity of becoming saturated with water. As it is from the lower part that the acid is again taken to be volatilised, and then sent on to the compressor, the aspirated acid is always relatively pure. The condenser of the sulphurous acid machine thus acts as a sort of purifier; it is only necessary to be able, from time to time, to get rid of the water which accumulates. On the other hand, all this will only take place when the apparatus is well filled; if the machine contains too little sulphurous acid, the separation will certainly take place in the condenser, but the water will be re-aspirated at the same time as the acid, and the corrosion of the iron will be effected very easily, especially if the compression takes place at a temperature above 70°.

M. Harpf, in No. 21 of the *Zeitschrift für Angewandte Chemie*, has criticised my communication and rectified a small error which had accidentally crept in. He also draws attention to the equation I gave to explain the phenomenon of the solution of the iron in sulphurous acid containing water:—$2Fe+3SO_2=FeSO_3+FeS_2O_3$.

It is evident that Schützenberger's explanation, that ferrous hydrosulphite is first formed and then turns into hyposulphite, is true in the case of the attack with commercial acid, since he recognises the necessity of water for the reaction which takes place, I have never been able to detect hydrosulphurous acid, but it must be remembered that this acid decomposes by heat, and my experiments have only been carried out at a fairly high temperature. The hypothetical hydrosulphite of iron is in any case decomposed before the cylinder is emptied and re-heated, but it is necessary during the experiment at a temperature higher than 70° that it be formed and decomposed into hyposulphite at the same time. Even in the product of the reaction, taken from the ice machine above mentioned, there was no hydrosulphurous acid, although the substance had not been re-heated. Finally, the supposition that the yellow colour of the pure sulphurous acid first used is caused by the presence of hydrosulphurous acid, is not altogether legitimate. It is certainly not inadmissible that commercial sulphurous acid contains this material, but then the cylinders and receptacles would be attacked, even at a low temperature, and this fact would decidedly complicate the conditions of transport of the pure acid. I therefore think that, considering we have not proved the existence of hydrosulphurous acid in the sulphurous acid, it is preferable to attribute the accidental yellow colouration of the latter to the presence of lubricating oil.—*Zeitschrift fur Angewandte Chemie*, 1899.

THE PRODUCTION OF IODISED POTASSIUM AND AMMONIUM CARNALLITES.

By A. DE SCHULTEN.

THESE carnallites are obtained if, in the preparation of the bromised carnallites already described (*Bull. Soc. Chim.*, 1897, [3], xvii., 166), we replace the bromides by equivalent quantities of iodides.

To prepare the iodised potassium carnallite, we abandon a mixture of a solution of 8 grms. of iodide of potassium and 200 grms. of the salt MgI_2+6H_2O, to slow evaporation in vacuo over sulphuric acid. The crystals, dried between blotting-paper, and left over sulphuric acid, gave on analysis the following figures, leading to the formula KI,MgI_2+6H_2O:—

	Found.	Calculated.
K	7·34	7·09
Mg	4·36	4·42
I	69·00	68·93
H_2O	19·43	19·56
	100·13	100·00

The water was estimated by calcining the material with a large excess of oxide of lead.

Iodised carnallite occurs in flat prisms, having, like the bromised carnallite, the facets *p* (001), and *m* (110). The angles are 90° or thereabouts. The crystals are very hygroscopic, so I have not been able to measure the angle accurately.

The density of the crystals is 2·547 at 15°. Their molecular volume is therefore 217.

The iodised ammonium carnallite is obtained by the evaporation, in vacuo over sulphuric acid, of a solution containing 15 grms. of iodide of ammonium and 125 grms. of the salt MgI_2+6H_2O. The analysis of the crystals gave the following figures, leading to the formula NH_4I,MgI_2+6H_2O:—

	Found.	Calculated.
NH_4	3·39	3·40
Mg	4·64	4·59
I	71·46	71·67
H_2O	20·19	20·34
	99 68	100·00

The water was determined in the same manner as with the corresponding bromised compound (*loc. cit.*).

The appearance and crystallographic properties of iodised ammonium carnallite are the same as those I have described for the iodised potassium carnallite. The crystals are very hygroscopic.

The density of the crystals is 2·346 at 15°. Their molecular volume is therefore 226.—*Bull. Soc. Chim.*, Series 3, xxiii., No. 5.

The Hydrates of Chloride of Magnesium.—A. Bogorodsky.—The author confirms the existence of the hydrate with $12H_2O$ announced by van't Hoff and Meyerhoffer, and describes its interesting properties. A solution containing $MgCl_2+12·06H_2O$ cooled down to −17° rapidly gives very beautiful crystals of the hydrate, which appear to be quadratic. They are lighter than the liquor in which they form. The crystallation takes place with augmentation of volume, and is in every respect comparable to the congelation of water. The fusing point of this hydrate is −16°.—*Journ. Soc. Phys. Chim. R.*, xxx., 1·· 735·

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, April 27th, 1900.

Mr. T. H. Blakesley, Vice-President, in the Chair.

This meeting, by invitation of Sir Norman Lockyer, F.R.S., was held in the Solar Physics Observatory, South Kensington.

Sir Norman Lockyer gave "*A Short Account of the Physical Problems now being Investigated at the Solar Physics Observatory and their Astronomical Applications.*"

The chief work carried on at the Observatory is the comparison of stellar spectra with spectra obtained from lights emitted by laboratory sources. The light from a star (or the sun), and from an arc (or a spark), are focussed alternately upon the slit of a spectroscope, and the two spectra are photographed side by side upon the same plate. The number of lines in the arc spectrum depends upon which part of the arc is focussed on the slit. The image of the centre is rich in lines, the image of the edge gives a few single lines. Changes in spectra are also dealt with. The thickening and thinning of lines depends upon several things. In the first place, it depends upon the density of the substance, and thus the hydrogen lines in the spectrum of Sirius are much broader than those in α Cygni, the hydrogen being denser in the former star. Changes may also be produced by variations in quantity. A reduction in the quantity of a substance generally simplifies its spectrum, the longest line disappearing last. The motion of a luminous body to or from the spectroscope alters the wave-length of the light emitted, and produces a shift in the lines of the spectrum. The amount of deviation is a measure of the velocity of approach. In the case of Nova Aurigæ we have dark and bright lines of the same substance side by side. This shows that there are two bodies involved, moving with different velocities, the one giving a radiation, and the other an absorption spectrum. Another change in the lines depends upon temperature. In general, an increase in temperature produces a greater number of lines, a notable exception being sodium, which gives its full number of lines at the temperature of an ordinary Bunsen flame. The spectra of metals obtained from the arc and by sparking are often quite different. Those lines which make their appearance or are intensified in passing from the arc to the higher temperature of the spark are known as enhanced lines. The comparison of stellar spectra with laboratory spectra is often easy. For instance, the presence of iron in the sun, and hydrogen in Sirius is easily seen. Several lines in the spectrum of Bellatrix have been shown to be due to helium, the position of the lines being exactly the same as those due to the gases from clevite. In many cases it is possible to build up the spectrum of a star from the spectra of its constituents taken at the proper temperatures. For instance, the spectrum of γ Orionis can be closely imitated by means of oxygen, nitrogen, and carbon, together with the well-marked lines of hydrogen and helium. We can roughly estimate by the character of the spectra of stars, the temperature of those stars, and thus arrive at a stellar thermometry. Starting with a hot star like Bellatrix, and passing through β Persei, γ Lyræ, Sirius, Castor, Procyon, to Arcturus, a cold star, we have a gradual change in the character of the lines which appear in the spectrum of any constituent. The widening of the lines in the case of spectra of sun-spots enables us to trace changes in temperature of the sun, and we can compare these temperature changes with a variety of terrestrial phenomena, such as variation in latitude. The extraordinary number of lines exhibited by many metals suggests that what we are accustomed to call chemical elements are really complex bodies, which are made up of simpler ones. Attempts have been made to build up the spectra of metals by superimposing simple sets of lines upon one another. In many cases a great number of series would be required to represent things completely. In the case of hydrogen it would be necessary to have at least 27 series to give the structure spectrum only. Taking the atomic weight of hydrogen as unity the atomic weight of the little masses, which might give rise to any one of the series, would be about 0·0019. This is of the order of magnitude of the small bodies whose existence has been suggested by Professor J. J. Thomson from his work on ions.

NOTICES OF BOOKS.

A Manual of Chemistry, Inorganic and Organic, with an Introduction to the Study of Chemistry. By A. P. Luff, M.D., B.Sc., F.I.C., and F. J. M. Page, B.Sc., F.I.C., &c. Illustrated with 40 Engavings. London: Paris, New York, and Melbourne: Cassell and Co., Lim. 1900. Pp. 541.

Although not so announced on the title-page, we find that this book is intended principally as a guide to the study of chemistry for the use of medical students. There is no doubt that chemistry and medicine are becoming more closely allied every year, and the physician is becoming more and more dependent on the chemist (not the *druggist*), especially in the matter of new organic compounds, the uses of which are so rapidly spreading in medicine.

Organic chemistry having become so vast a science, the student of medicine is apt, on the one hand, to find himself out of his depth, so to speak, in attempting to master the contents of the larger handbooks on the subject, while, on the other hand, many of the smaller works omit a good deal of matter which is essential to his ultimate success in medicine. It is to correct these two extremes that the authors have compiled this volume, bearing constantly in mind the special wants of the medical student, while at the same time ensuring to the careful reader a thorough grounding in the fundamental principles of modern chemical science.

Curiosities of Light and Sight. By Shelford Bidwell, M.A., LL.B., F.R.S. With fifty illustrations. London: Swan, Sonnenschein, and Co., Limited. 1899. Pp. 226.

The essays and lecture-notes which form the ground-work of this volume treat the subject in a popular and informal manner, which makes very pleasant reading. There is in fact no other easily accessible account of many of the curious matters touched upon. A perusal of this volume shows either what a very imperfect organ the human eye is, or else what very bad use we make of it. There is no wonder that the conjurer can make us see what he wishes us to see, when we find our eyes so easily deceived by straightforward experiments in which no deception is attempted or intended.

The author begins with an examination of the theory of light and its action on the eye. The essential nature of the action exerted by ether waves is still undetermined, though the old belief that the sensation was produced by a series of mechanical beats similar to the action of air-waves on the ear no longer carries any weight. Ether waves can bring about chemical changes as in photography; they can also produce electrical effects as, for example, when they are allowed to fall on a piece of prepared selenium; but it is inconceivable, and, further, there is no evidence that they are able to impart their own vibrations of such enormous frequency to any part of the human organism; many guesses at the truth have been made, but it is most likely that the effect is both electrical and chemical.

Again, the range of sensibility of different eyes may vary in the same manner as do different ears, and it is by no means probable that all animals, and especially insects, have the same limits of vision as man; further, the same vibrations falling in different places will give very different results. A chemical action will be caused by the ray falling on the leaf of a plant, an electrical action if it falls on a selenium cell, and the sensation of light if it falls on an eye.

In the portion of the book dealing with colour and its perception a great deal of interesting and little known information is to be found; while the chapters on optical illusions and curiosities of vision are most attractive, and will be read with enjoyment.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxi., No. 24.

Binoxide of Molybdenum.—M. Guichard.—Already inserted.

Estimation of Sulphuric Acid in the presence of Iron.—G. Wyrouboff.—A controversial paper, not suitable for abstraction.

Antimonic Acids and Antimoniates.—A. E. Delacroix.—Already inserted.

Action of Sulphate of Methylene on Benzylic Alcohol.—Marcel Delépine.—For the safe utilisation of the action of sulphate of methylene on benzylic alcohol with the object of preparing the formol and the acid sulphuric ether of this alcohol, it is necessary to strictly adhere to the following rules:—The sulphate must be mixed with five or six parts of benzylic alcohol and slowly heated to 60—65° with continuous stirring. The temperature must not be allowed to surpass 70°, or the reaction will be so violent that the benzylic alcohol will instantly become resinous, and the temperature rise to over 300°. When all the sulphate is dissolved at the lower temperature, a pale yellow liquid results; agitation with water removes the greater part, but not the whole, of the acids formed. The acid liquors, neutralised with carbonate of barium, give dissolved benzylsulphate of barium. The still acid insoluble portion must not be distilled directly, for, at a certain moment, a violent reaction occurs, which carbonises everything, and the temperature rises to over 360°. To prevent this, the insoluble oil must be diluted with one volume of ether and neutralised with carbonate of potassium; a precipitate of bicarbonate, sulphate, and benzylsulphate of potassium is formed, from which the latter is extracted with boiling absolute alcohol. As for the ethereal solution, it is distilled *in vacuo* and fractionated; the benzylic alcohol passes over first, then the benzylic formol. Benzylic formol, $CH_2(OC_7H_7)_2$, is a colourless liquid, which does not solidify at $-23°$; it boils at about 210—220° under 20 m.m., and at 330° at the ordinary pressure. By evaporating the solution of benzylsulphate of barium *in vacuo*, it separates in long colourless needles having the formula $(C_6H_5.CH_2.O.SO_3)_2Ba+2H_2O$. Benzylsulphate of potassium crystallises in absolute alcohol in fine pearly needles; its proportion of potassium gives the anhydrous salt the formula $C_6H_5CH_2.O.SO_3K$.

Preparation of Azelaïc Acid.—M. Maquenne.—Castor oil is saponified by heating for a few minutes with an alcoholic solution of potash and diluting with warm water; the fatty acids are precipitated with a slight excess of sulphuric acid, decanted, washed, and dried. 30 grms. of the raw ricinoleic acid thus obtained are dissolved in 200 c.c. of a 4 per cent solution of potash and gently heated, then poured suddenly into a 2-litre flask containing 1 litre of warm water (at about 35°) and 75 grms. of permanganate of potassium. When all the permanganate is dissolved, the flask is heated on the water-bath at 90° until the red colour entirely disappears, when 90 grms. of 50 per cent sulphuric acid are gradually added. The precipitate is filtered and washed with warm water; all the liquors are collected and made up to about 500 c.c. On cooling, the azelaïc acid crystallises out in transparent flakes.

On α-Dimethylisocrotonic Acid (2-Dimethylbutinoïc-3).—L. Bouveault.—The ethers of the non-saturated αβ-acids condense easily with the soda derivatives of the malonate or the acetylacetate of ethyl. The author has tried whether the ethers of the non-saturated βγ-acids have the same property. He therefore tried α-dimethylisocrotonic ether,—

$$CH_2=CH-\overset{CH_3}{\underset{CH_3}{C}}-CO_2C_2H_5,$$

prepared by the dehydration of the ether-alcohol. It was expected that the reaction would lead to α-dimethyladipic acid, or to an ααβ-trimethylic glutaric acid; there was, however, no condensation produced at all. It therefore seems that, for this interesting reaction to take place, the double bonds should be immediately adjoining a negative group. Some interesting results were, however, obtained, such as the preparation of the dimethyl-β-oxybutyrate of ethyl.

Rhamninase and Xanthorhamnine.—C. and G. Tanret.—For examining the activity of rhamninase, the authors prepared solutions of xanthorhamnine (at one-fifteenth), and added equal weights of rhamninase; that is, 0·165 grm. per 100 c.c. of solution containing 6·66 grms. of glucoside. After four hours and ten minutes in the oven at the desired temperature, the precipitates were collected, washed, and dried at 100°. The results obtained are given in the following table. T = the temperature of the experiment; *t*, the time when the precipitates commenced to deposit; P, the weights of these precipitates; and *p*, these weights in proportion to that formed at 70°, which was taken as 100.

T.	*t*.	P.	*p*.
	Minutes.	Grms.	
85°	0	0	0
80	28	0·498	31·5
75	6	1·245	78·8
70	7	1·580	100·0
65	10	1·427	90·3
60	20	1·321	83·6
55	25	1·202	76·1
50	33	1·183	74·8
45	35	1·173	74·2
40	38	0·629	39·8
35	40	0·466	29·5
30	40	0·396	25·0
25	72	0·200	12 6
20	225	P too slight to collect.	

Saponification of Benzonitrile.—Ch. Rabaut.—By heating an alcoholic solution of benzonitrile with weak potash in a flask fitted with a vertical condenser, the author obtained benzamide in almost theoretical quantity. The return of benzamide is, in fact, a function of the quantity of potash used. If concentrated potash is used, benzoic acid is almost uniquely obtained. Under the same conditions, in the presence of potash, cyanide of benzyl gave the corresponding amide, but in less proportion.

Preparation of Anisic Aldehyde.—H. Labbé.—One part of anethol is added to 2 parts of glacial acetic acid in a large flask, and 3½ parts of nitric acid at 14 B. are

run in, taking care to put a few fragments of pumice at the bottom of the flask. The flask must be gently heated and shaken from time to time. After the oxidation is completed (about half an hour), the insoluble part is separated by decantation by means of a stoppered funnel, the aqueous portion is saturated with carbonate of soda; this separates an oil which is added to the first. This oil is agitated with bisulphite of soda; the crystallised magma, dried by means of a filter-pump, is washed with alcohol and ether. The preparation is then finished in the ordinary way. The return by this method is 69 or 70 per cent of theory.

Fixation of Acid Sulphite of Sodium on Double Junctions. — H. Labbé. — The fixation of a radical SO_3MH (M being an alkaline or alkaline-earthy metal) on a double junction, CH=CH, such as $\begin{matrix}-CH-CH_2\\ |\\ SO_3M\end{matrix}$ or $\begin{matrix}CH_2-CH-\\ |\\ SO_3M\end{matrix}$, has not been closely examined up to the present. The author has undertaken the examination of the subject with regard to different characteristic functions and different classes of compounds (aliphatic, aromatic, or turpenic). His first experiments have largely confirmed the hypothesis that the characteristic function of the molecule should have an influence on the greater or less facility with which double junctions of the molecule fix the acid sulphitic radical; while later experiments show that the phenomenon is governed by three principal factors — the nature of the compound, its characteristic function, and its solubility in the aqueous medium.

Genesis of the Terpenic Constituents of Essence of Bergamot.—C Charabot.—The author finds that during the maturing of the fruit of the *Citrus bergamia*, the composition of the essential oil is modified in the following manner:—1. The proportion of the free acids diminishes slightly. 2. The richness in acetate of linalyl increases. 3. The proportion of free linalol, and even of total linalol, diminishes in a very marked manner. 4. The quantity of the terpenes augments, without the relative proportion of the two carbides being modified. 5. The proportion of bergaptene diminishes. To sum up, the active period of the formation of the linalol is that corresponding with the development of the fruit, the etherification accompanied by dehydration of this terpenic alcohol takes place mainly during the maturing of the fruit.

MISCELLANEOUS.

Royal Institution.—The Annual Meeting of the Members of the Royal Institution was held on May 1st; Sir J. Crichton-Browne in the Chair. The Annual Report of the Committee of Visitors for the year 1899, testifying to the continued prosperity and efficient management of the Institution, was read and adopted, and the Report on the Davy Faraday Research Laboratory of the Royal Institution, which accompanied it, was also read. Sixty-three new Members were elected in 1899. Sixty Lectures, Seventeen Evening Discourses, and two Centenary Commemoration Lectures were delivered in 1899. The Books and Pamphlets presented in 1899 amounted to about 280 volumes, making, with 672 volumes (including Periodicals bound) purchased by the Managers, a total of 952 volumes added to the Library in the year. Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committee of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year. The following gentlemen were unanimously elected as Officers for the ensuing year:—President—The Duke of Northumberland; Treasurer—Sir James Crichton-Browne; Secretary—Sir William Crookes; Managers—Sir Frederick Abel, Bart., Sir Frederick Bramwell, Bart., Dr. Thomas Buzzard, Sir William James Farrer, Mr. Hugh Leonard, The Right Hon. Lord Lister, Mr. Frank McClean, Mr. Raphael Meldola, Dr. Ludwig Mond, Sir Andrew Noble, Mr. Alexander Siemens, Mr. Basil Woodd Smith, Mr. William Hugh Spottiswoode, The Hon. Sir James Stirling, and Sir Henry Thompson, Bart.; Visitors—Dr. Charles Edward Beevor, Mr. William Henry Bennett, Mr. W. A. B. Burdett-Coutts, Mr. Joseph G. Gordon, Mr. Maures Horner, Sir Alexander Campbell Mackenzie, Mr. Henry Francis Makins, Mr. Carl Edward Melchers, Mr. John Callander Ross, Mr. William James Russell, Mr. Alan A. Campbell Swinton, Sir James Vaughan, Mr. John Jewell Vezey, Colonel H. Watkin, and Mr. James Wimshurst.

A Hydrated Bromide of Copper and Lithium.—N. Kournakoff and A. Sementchenko.—By evaporating on the water-bath a solution of $CuBr_2$ and $2BrLi$ until the appearance of crystals, and then allowing to cool while protected from moisture, we obtain black needles, appearing bronze by reflected light, excessively hygroscopic, having the formula $CuBr_2.2(LiBr_3H_2O)$. The authors admit that the water of crystallisation is fixed to the lithic and not the cupric salt.—*Journ. Soc. Phys. Chim. R.*, xxx. 701.

MEETINGS FOR THE WEEK.

Monday, 7th.—Society of Arts, 8. (Cantor Lectures). "The Incandescent Gas Mantle and its Use," by Prof. Vivian B. Lewes.
—— Royal Institution, 5. General Monthly Meeting.
Tuesday, 8th.—Royal Institution, 3. "Studies in British Geography," by Hugh Robert Mill, D.Sc., LL.D., &c.
—— Society of Arts, 8. "Art Metal Work," by Nelson Dawson.
Wednesday, 9th.—Society of Arts, 8. "Improvement of our Roads," by A. Moresby White.
—— Royal. (Conversazione).
—— and Thursday, 10th.—Iron and Steel Institute, 10.30. Annual Meeting.
Thursday, 10th.—Royal Institution, 3. "A Century of Chemistry in the Royal Institution," by Prof. Dewar, M.A., LL.D., F.R.S.
Friday, 11th.—Royal Institution, 9. "Shakespeare and True Patriotism," by Sidney Lee.
—— Physical, 5. Discussion of Prof. Lodge's Paper "On the Controversy concerning Volta's Contact Force." "Heat of Formation of Alloys," by J. B. Taylor. "Want of Uniformity in the Action of Copper-Zinc Alloys on Nitric Acid," by Dr. Gladstone, F.R.S. An Electromagnetic Experiment, and Experiments illustrating the Aberration called Coma, by Prof. S. P. Thompson, F.R.S.
Saturday, 12th.—Royal Institution, 3. "South Africa—Past and Future," by Dr. Alfred P. Hillier.

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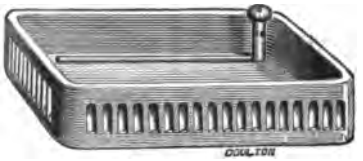

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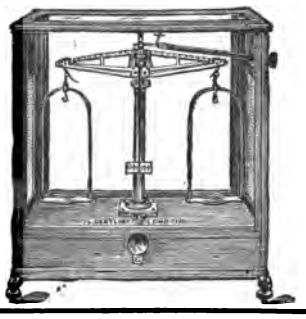

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VOL. LXXXI., No. 2111.

THE KINETIC THEORY OF PLANETARY ATMOSPHERES.*

By G. H. BRYAN, Sc.D., F.R.S.

THE application of the kinetic theory to the atmospheres of planets dates from the paper of Waterston, who gave an investigation based on the then only possible assumption of equal velocities for all molecules, an assumption since known as Clausius's law. Of later papers reference is due in especial to Dr. Johnstone Stoney's memoir "Of Atmospheres on Planets and Satellites" (*Trans. R. Dublin Soc.*), in which the test of permanence of a gas in the atmosphere of a planet is made to depend on the ratio of its velocity of mean square to that relative velocity which would enable a suitably projected body to escape from the planet's attraction. If it be admitted, as Dr. Stoney assumes, that helium cannot exist in our atmosphere, it follows that vapour of water cannot exist on Mars.

The author's object has been to investigate the logical conclusions obtained by applying the Boltzmann Maxwell distribution to the atmospheres of planets. In 1893 calculations were made, having special reference to the absence of atmosphere from the Moon, but these took no account of axial rotation. When this cause is taken into account, the distribution of co-ordinates and *relative* velocities of the molecules is found to be the same as if the planet were at rest, and "centrifugal force" applied to the system. The surfaces of equal density are of the forms originally investigated by Edward Roche, of Montpellier, and they cease to be closed surfaces when passing to the outside of the point on the equatorial plane where centrifugal force just balances the planet's attraction. Calling the surface through this point the "critical surface," the density of molecular distribution over this surface must be very small to ensure permanence. The ratio of the density at the planet's surface to the density at the critical surface has been called the "critical density ratio," and the author calculates its logarithm for particular gases at different temperatures on the various planets. The use of this logarithm has the advantage that the calculation can at once be extended to any gas at any temperature.

The high value obtained in the case of helium considered in reference to the earth, appears to afford abundant proof that if helium existed in our atmosphere it would possess a very high degree of permanence at ordinary temperatures. To test this point further, a calculation is made of the total rate at which molecules would flow across the critical surface, this rate being regarded as a superior limit to the rate at which the planet would lose its atmosphere, since it takes no account of molecules which describe free paths beyond the limit and fall back again. To further exhibit the results in a tangible form, the rate of flow is estimated by the number of years in which the total amount of gas escaping across the critical surface would be equal to the amount of the gas in a layer covering the surface of the planet to the depth of 1 c.m. This measure is independent of the actual quantity of the gas under consideration existing in the atmosphere, since, if this quantity be increased, the rate of flow across the critical surface and the amount of gas present in the surface layer 1 c.m. thick will be increased in the same proportion.

If a gas of molecular weight 2, such as helium, be supposed to exist in the earth's atmosphere, the loss in question would occupy $3{\cdot}5 \times 10^{28}$ years at $-73°$ C., 3×10^{19} years at 27° C., $8{\cdot}4 \times 10^{10}$ years at 127° C., 6×10^{4} years at 227° C., and 222 years at 327° C.

If we halve the absolute temperatures we have the conditions applicable to hydrogen, the losses in question therefore taking place in $8{\cdot}4 \times 10^{10}$ years at $-73°$ C., 6×10^{4} years at $-23°$ C., and 222 years at 27° C.

For water vapour on Mars, the corresponding results are $1\,2 \times 10^{28}$ years at $-73°$, $1{\cdot}9 \times 10^{16}$ years at 27° $2{\cdot}4 \times 10^{9}$ years at 127°, $4{\cdot}3 \times 10^{4}$ years at 227°, and 106, years at 327°.

These figures indicate that helium cannot practically escape from our atmosphere at existing temperatures, nor can vapour of water escape from the atmosphere of Mars. A leakage may and undoubtedly does take place which may appear considerable when estimated by the number of actual molecules escaping, but it is wholly inappreciable relative to the mass of gas left behind.

At a future time I propose to examine the corresponding results, based on the hypothesis that the atmosphere of a planet is distributed according to the adiabatic instead of the isothermal law.

* Abstract of a Paper read before the Royal Society, April 5, 1900.

ON THE DISCOVERY AND OCCURRENCE OF MINERALS CONTAINING RARE ELEMENTS.*

By Baron A. E. NORDENSKIÖLD.

THE first mineral referred to is scheelite, and the next cerite, which contains no less than four rare metals. The incandescent light produced when the latter mineral is fused with charcoal powder was first observed by Cronstedt in 1751. The discovery of glucina, lithia, selenium, and yttria is next referred to. Minerals containing yttria and oxides related to it were, at one time, thought to be almost limited to certain pegmatite-veins running in a broad zone on both sides of the 60th parallel of latitude. Latterly, fluocerite, orthite, and gadolinite have been found in Dalecarlia; and among these minerals Benedicks discovered a silicate of yttrium containing 1·5 per cent of nitrogen and helium. The author discovered kainosite, a silico-carbonate of yttrium and calcium, among minerals from Hitterö; and the same mineral was subsequently discovered in the flucan, fissures, and drusy cavities at the Nordmarken mines. The last mentioned discovery and others related to it appear to suggest that the mode of formation of fissure-minerals is not so unlike that of the pegmatite-veins of the Primary rocks as is generally supposed.

Thorium, discovered by Berzelius in 1829, was originally obtained from the rich mineral-locality of Langesund (called Brevig in mineralogical literature), but it has since been recorded from other localities, including Arendal and Finnish Lapland. It is now obtained from the monazite-sand of rivers in the Brazils and South Carolina. Thorite contains about 0·5 per cent of inactive gas, probably a mixture of nitrogen and helium; but the latter element was first obtained from the mineral cleveite, also containing thorium, discovered by the author in 1877. Other minerals bearing nitrogen, argon, or helium are referred to; and under the head of minerals bearing tantalum, mention is made of Giesecke's discoveries in Greenland. Among these is fergusonite, one of the richest sources hitherto known for obtaining that mysterious gas, or mixture of gases, which on our planet seems to be almost exclusively confined to minerals containing rare earths. "The group of earths, as well as the group of gases, of which we are here speaking, might therefore be compared with certain genera among organic beings, whose species, having not yet fully differentiated, offer to the descriptive zoologist or botanist difficulties analogous to those with which chemists meet in endeavouring to separate the rare earths and rare gases."

* Read before the Geological Society of London, April 4th, 1900.

Discussion.

Prof. Miers remarked on the isolated occurrence of the rare elements in certain patches of the earth's surface, and deplored the ignorance which at present prevails concerning their distribution in Cornwall, where rhabdophane, churchite, and monazite have been discovered; monazite he had himself found in the slate at Tintagel. The interesting historical survey given in the present paper directed attention to the importance of the mineralogical distribution of these rare elements.

THE PREPARATION OF FLUORINE BY ELECTROLYSIS IN AN APPARATUS OF COPPER.

By HENRI MOISSAN.

Up to the present time fluorine has been obtained by the electrolysis of a hydrofluoric solution of fluoride of potassium in a platinum apparatus. Shortly after the commencement of our research we announced that the platinum of the electrodes and of the apparatus was attacked, that a certain quantity of this metal went into solution, and that from this moment the electrolysis became more regular (*Ann. Chim. Phys.*, [6], xxiv., p. 224).

The use of platinum, and the wearing of the electrodes and of the vessel, which was fairly rapid, made this apparatus very expensive.

To find out whether it was possible to replace the platinum by another metal we placed samples of various metallic wires at the bottom of the electrolytic apparatus and proceeded with the preparation of the fluorine in the usual manner. We observed that, of the different metals used in these experiments, the one that was attacked the least was copper, that is, if the acid was perfectly free from water. This fact further agrees very well with the researches made by M. Poulenc on the properties of fluoride of copper (*Ann. Chim. Phys.*, [7], xix., p. 66).

In view of these preliminary experiments we constructed a copper U-tube, of as near as possible the same shape as our platinum electrolyser. Its volume was a little greater, being of about 300 c.c. capacity, and easily permitting the electrolysis of 200 c.c. of hydrofluoric acid, made conducting by means of 60 grms. of hydrofluorate of fluoride of potassium. The method of closing the apparatus was the same, but the shape of the electrodes was changed. In our previous experiments we made use of cylindrical rods of platinum the ends of which were clubbed; wishing to have a greater surface we gave the electrodes the form of open hollow cylinders. The surface was increased so as to get a higher return. The electrodes were still of platinum, as for this part of the apparatus we were unable to use copper.

In some experiments made with copper electrodes the metal went into solution as soon as the electrolysis began, and a badly conducting layer of fluoride of copper, which stopped the current, was quickly deposited on the positive electrode. If the mixture of hydrofluoric acid and fluoride of potassium is entirely free from water, the electrolysis goes on very satisfactorily with platinum electrodes in a copper vessel. Under these conditions this latter will not be attacked; it is probable that the fluorine which soon goes into solution in the hydrofluoric acid forms on the surface of the copper a thin layer of this protecting fluoride, of which we have previously spoken, insoluble in hydrofluoric acid.

The return from this apparatus was found by measuring the volume of the hydrogen given off at the negative pole in a certain time. In a series of preliminary experiments we satisfied ourselves that the volume of oxygen produced by the action of the fluorine on the water corresponded with the volume of hydrogen given off at the positive pole, allowing, of course, for the amount of ozone formed.

With a current of 50 volts and 15 ampères we obtained a return of about 15 litres per hour when the experiment lasted about from six to ten minutes. By using a current of 20 ampères at the same voltage the return was increased to 8 litres, but in the second case the experiment could not be continued for long owing to the heating of the liquid, and, in spite of an artificial cooling of $-50°$, the fluorine gas carried off abundant fumes of hydrofluoric acid.

It is also of importance not to lower the temperature too much, or there will be a considerable formation of a compound of hydrofluoric acid with the alkaline fluoride. This new copper apparatus gave very good results in experiments lasting several hours, and we were able to examine several new questions, for which a continuous current of fluorine was necessary.—*Bull. Soc. Chim.*, Ser. 3, xxiii., No. 8.

RAPID METHOD FOR THE ESTIMATION OF CLAY IN SOILS.

By F. POQUILLON.

The usual method adopted for making this estimation is a very tedious one; by its means we cannot estimate the clay in less than eight or ten days.

The method I am about to describe will enable this estimation to be done in two or three days at the most. It is based on the fact that if, instead of mixing the earth with distilled water, we use a dilute solution of hydrochlorate of ammonia, the clay is held in suspension while it is at the same time coagulated, thus enabling the sand to deposit almost immediately.

Ten grms. of the soil are placed in a small porcelain crubible and rubbed round the sides with the first finger while adding water, drop by drop, until about 25 c.c. has been added. This mixture is transferred to a beaker of 150 c.c. capacity, and about 100 to 120 c.c. of a solution of hydrochlorate of ammonia, at 1 grm. per litre, are added. After well stirring with a glass rod, it is allowed to settle for five minutes, and the liquid decanted into a 1 litre beaker. On this residue 100 to 125 c.c. more of the hydrochlorate solution are poured; stir well again, allow to stand for five minutes, and decant into the 1 litre beaker. This operation is repeated until the wash-waters are quite clear: this will require six or eight washings for the most argillaceous soils. The residue is treated with hydrochloric acid diluted with water, washed with distilled water, and dried; its weight gives the total sand.

The thick liquid remaining in the 1 litre beaker is treated with a few drops of hydrochloric acid, to decompose the calcareous matter and complete the coagulation of the clay. It is then allowed to stand until the supernatant liquid is clear; this requires two or three hours. The clay which deposits is collected on a weighed filter, washed with distilled water, dried, and weighed.

This method has the following advantages over the old one:—

1. Instead of making two washings on the filter, one to remove the lime and chalky matter and the other to free the clay from the hydrochlorate of ammonia, a single washing enables us to get rid of both the lime and hydrochlorate at the same time.

2. Instead of obtaining the clay in suspension in 4 or 5 litres of water, it is now found in 750 to 1000 c.c.: this is in one-fifth or in one-sixth of the volume.

3. Instead of getting the clay on the filter after five or six days, this stage is reached after about three hours.

4. The clay obtained by this process is washed more rapidly than that obtained by the old method (I have never taken more than a day and a half to effect the same amount of washing that used to require three or four days by the old method).

Finally, the result obtained by these two methods, used simultaneously, are absolutely comparable, as the following figures will show :—

Clay, per Thousand Parts of Soil.

Old method = 206·0, 60·2, 198·0, 218·0, 122·0, 129·3.
New method = 205·8, 60·5, 198·0, 217·1, 121·9, 129·5.

—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 4.

THE PRODUCTION OF VANADINITES OF CADMIUM.

By A. de SCHULTEN.

In a paper on the phosphates and arseniates of cadmium (*Bull. Soc. Chim.*, Series 3, vol. i., p. 472) I showed that this metal forms apatites and arsenio-apatites. That being so, I tried whether cadmium would also form vanadinites, and I obtained, as result, a chlorovanadate and a bromovanadate of cadmium belonging to the family of vanadinites.

Chlorised Vanadinite of Cadmium.—To prepare this body we melt 50 grms. of chloride of cadmium with 4 grms. of carbonate of cadmium and 1·4 grms. of vanadic anhydride. On cooling the melted mass, very fine needles of vanadinite of cadmium are formed, which are separated by washing with water. By diminishing the quantity of chloride of cadmium in this experiment, we can get a slightly more voluminous crop of crystals, but they are contaminated with impurities. These crystals can be purified by treating them with dilute acetic acid. But the crystals will, in any case, be slightly attacked by this treatment. The analysis of these crystals gives the following figures, which lead to the formula $3Cd_3.(VO_4)_2.CdCl_2$:—

	Found.	Calculated.
Cl	3·69	3·76
Cd	59·95	59·51
V_2O_5	29·01	29·08
O	7·35 (by diff.)	7·65
	100·00	100·00

The crystals dissolve easily in weak acids. When heated to redness they melt, giving off fumes of chloride of cadmium. In the cooled mass prismatic crystals can be seen, which are probably regenerated vanadinite.

Chlorised vanadinite of cadmium occurs in long hexagonal prisms. They are negative, and their density is 5·264 at 15°.

Bromised Vanadinite of Cadmium.—This body is obtained if, during the preparation of chlorised vanadinite just described, we replace the chloride of cadmium by the same quantity of bromide of cadmium. Bromised vanadinite of cadmium crystallises more readily than the chlorised vanadinite. The analysis of the crystals gave the following results, leading to the formula $3Cd_3(VO_4)_2.CdBr_2$:—

	Found.	Calculated.
Br	7·59	8·10
Cd	56·98	56·83
V_2O_5	27·48	27·77
O	7·95 (by diff.)	7·30
	100·00	100·00

The chemical, crystallographical, and optical properties of bromised vanadinite of cadmium are the same as those described for the corresponding chlorised compound. Its density is 5·456 at 15°.

By fusing carbonate of cadmium and vanadic anhydride with a large excess of iodide of cadmium, I obtained a small quantity of crystals having the appearance of the above-described vanadinites. These crystals, which contain iodine, vanadic acid, and cadmium, are probably iodised vanadinite of cadmium. However, I have not been able to remove the impurities present in these crystals without the latter being strongly attacked.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 5.

THE MEASUREMENT OF LOW TEMPERATURES.

By A. LADENBURG and C. KRUEGEL.

The authors have made use of a platinum-rhodium thermo-electric couple, and took three fixed points, viz.—the boiling-point of liquid air, −187°; the boiling-point of ethylene, −102·65°; and the sublimation point of a mixture of carbonic anhydride and alcohol, −76·6°. The temperature, t, was given a function of the number x of millivolts, by the equation—

$$t = -24{\cdot}948\,x + 1\,6744\,x_2 - 0{\cdot}2248\,x_3.$$

The correctness of this relation was controlled by taking the boiling-point of methane. The following results were obtained :—

	Boiling-point. °	Pressure. M.m.	Melting-point. °
Oxygen	−181·4	745	—
Nitric oxide	−141·4	757·2	−150
Ammonia	—	—	−77·05
Methane	−152·5	749	—
Ethane	−85·4	749	−171·4
Ethylene	−102 65	756·9	—
Propylene	−50·2	749	Remains liquid in liquid air.
Trimethylene	−35 about	749	−126
Acetylene	−83·8 subl.	—	—
Toluene	+110	—	−94·2
Ethylbenzene	+135—136	—	−94·2
Mesitylene	+164	—	−59·6
Cymene	—	—	−75·1
Chloride of methyl	—	—	−103·6
Bromide of ethyl	—	—	−116
Methylic alcohol	—	—	−94·9
Ethylic alcohol	—	—	−112·3
Ether	—	—	−112·6
Aldehyde	—	—	−120·7
Acetone	—	—	−94·9
Glycol	—	—	−17·4
Formate of methyl	+32—33	—	−101·3
Acetate of ethyl	—	—	−83·8
Ethylamine	+19—20	—	−85·2

Absolute alcohol, plunged into liquid air, congeals into an absolutely vitreous mass; if this is re-heated very slowly it forms a viscous substance, which if again cooled down slowly gives this time a crystalline opaque mass. Ether, acetone, and aldehyde crystallise directly.—*Berichte*, xxxii., p. 1818.

REDUCTION OF REFRACTORY METALLIC OXIDES BY ALUMINIUM.

By F. KUPPELWIESER.

In the accompanying table the author gives the results of his theoretical calculations relative to the possibility, from the thermo-chemical point of view, of applying Goldschmidt's process for the reduction of the metallic oxides by means of finely-divided aluminium (*Moniteur Scientifique*, March, 1899, p. 199). We thus see that the process is applicable in every case, with the exception of that of silica, where there is a deficit of 622 calories.

	For the production of 1 kilo. of—					
	Iron.	Manganese.		Silicon.	Chromium.	Tungsten.
Oxide used	Fe_2O_3.	MnO_2.	Mn_2O_3.	SiO_2.	Cr_2O_3.	WO_3.
Aluminium required kilos.	0·484	0·656	0·492	1·272	0·520	0·294
Heat developed calories	3459	4684	3512	9082	3713	2099
Heat absorbed by reduction	1796	2115	2000	7830	2200	1100
Heat for fusion of the slag	548	742	550	1439	582	324
" " metal	362	535	535	435	434	360
Total heat absorbed	2706	3392	3090	9704	3216	1784
Difference (possibly from radiation or other losses)	+750	+1299	+421	−622	+497	+315

In the experiments described by the author, the iron obtained was perfectly fluid, as was also the slag. The metal was sufficiently hot in itself to melt the submerged extremity of a bar of iron (10 m.m. diameter) in two minutes. It is, however, noteworthy that the metal thus obtained could not be worked hot, probably on account of the presence of dissolved oxygen. In the first experiment with manganese, pyrolusite was used; in the second, braunite. In both cases the metal obtained was perfectly fluid. According to Van der Welde, the fusing-point of manganese is 1900°; that of chromium is higher than that of platinum, and should be about 2200°.

The thermic data of tungsten are uncertain, and the margin of 315 calories is probably too small, since the reduction of tungstic acid was effected without difficulty. *Œsterr. Zeit. Berg- und Hüttenwesen*, 1899, p. 147.

ELECTROLYTIC DETERMINATIONS AND SEPARATIONS.*

By LILY G. KOLLOCK.†

Introduction.

That the electric current has greatly aided in the development of new and reliable methods for the determination and separation of metals is no longer questioned. That new material will continue to be added to what we now possess is also certain to occur. But in the old methods worked out long before correct ideas prevailed as to current density, concentration of the solution and electrode surface, gaps exist, and uncertainty must necessarily prevail in the minds of those who seek to reproduce the proper working conditions of these earlier methods. That the latter are in every sense satisfactory in the hands of those persons who are thoroughly acquainted with them is also beyond question. To render them, however, universally acceptable, and to render their place in analytical chemistry by electrolysis permanent, the absolute conditions for successful work on the part of any investigator must be definitely established. To this end, a number of the earlier methods in which the double cyanides were used as electrolytes have been carefully reviewed in the following pages, and the conditions of current density, ampèrage, voltage, and other factors fully worked out. Thus edited, doubt can no longer be thrown upon the methods proposed, and they will continue to be in their developed condition the most reliable for the purposes for which they have been suggested. They now for accuracy, neatness, and rapidity of execution most certainly excel the ordinary gravimetric and volumetric methods applied to the same metals.

Cadmium.

In 1878 Smith showed that cadmium could be precipitated quantitatively from its alkaline double cyanide solution. In the following experiments cadmium sulphate containing 0·1659 grm. cadmium in 10 c.c. was converted into the double cyanide, using 1 grm. of potassium cyanide. The solution was electrolysed with a current $N.D._{100}$ = 0·04—0·06 A and V = 2·9—3·2. The solution was heated to 60° C. After the current had passed for six hours, the deposit was washed with hot water and alcohol, dried, and then weighed. The solution poured out of the platinum dish, and examined qualitatively for cadmium, showed that all the cadmium had been deposited in this time. It was likewise entirely precipitated when the current was allowed to act on the solution through the night in the cold. The following results were obtained (see Table I.).

Silver.

It is stated (Smith's "Electro-chemical Analysis," p. 77) that silver may be precipitated from a cyanide solution containing an excess of potassium cyanide. It may be deposited with a low current, and has the advantage of non-precipitation of silver peroxide at the anode.

Accordingly a silver nitrate solution containing 0·1270 grm. of silver in 25 c.c., after the addition of 0·5 grm. of potassium cyanide, was diluted to 125 c.c. This solution was electrolysed with a current $N.D._{100}$ = 0·07 A and V = 3·2. The temperature was 65° C. The deposition was complete in three hours. With a current of $N.D._{100}$ = 0·1 A the silver was deposited completely in two hours. The relation between the current and the time factor may be seen in Table II.

Separations.

Silver from Platinum.—Smith (*Am. Chem. Journ.*, xiii., 417) described this separation. A current of 1 c.c. of oxy-hydrogen gas per minute acted upon a solution of the double cyanide produced by adding $2\frac{1}{2}$ grms. of potassium cyanide. The silver was deposited free from any trace of platinum.

The separation, therefore, was taken up in order to get the exact working conditions. The salts of the metals used were silver nitrate containing 0·1026 grm. of silver, and platinic chloride containing from 0·0879 grm. to 0·1758 grm. of platinum. To the combined solutions 1·25 grms. of potassium cyanide were added, and the whole diluted to 125 c.c. The current used was $N.D._{100}$ = 0·04 A and V = 2·5. The temperature was 70°. In from three to four hours the current was interrupted, and the deposit washed with hot water and alcohol. It was found to be free from platinum. The filtrate gave no reaction for silver. (See Table III.).

Silver from Copper.—In the *American Chemical Journal* (xi., 264) there appears a series of forty experiments relating to the separation of these metals. Copper was found to have been precipitated with the silver when the deposition took place from a cyanide solution. It was then thought that copper could not be separated from silver by this method. In 1889 a set of new experiments was reported. By lowering the strength of the current the separation was made. The silver deposit contained no copper, nor could silver be detected in the copper solution poured from the dish. In 1895 Smith and Wallace published a report of the separation made in that year.

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxi., No. 10.

† From the author's thesis for the degree of Ph.D.

TABLE I.

Cadmium. Grm.	Potassium cyanide. Grm.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time.	Metal found. Grm.
0·1659	1	125	$N.D._{100}$ = 0·06 A.	2·9	57°	7 hours	0·1657
0·1659	1	125	,, = 0·04 A.	2·9	Cold	During night	0·1650
0·1659	1	125	,, = 0·06 A.	3·2	60°	3 hours	0·1655

TABLE II.

Silver present. Grm.	Potassium cyanide. Grm.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1270	0·5	100	$N.D._{100}$ = 0·07 A.	3·2	65°	3	0·1271
0·1270	0·5	100	,, = 0·07 A.	3·2	65°	3	0·1270
0·1270	0·5	100	,, = 0·06 A.	3·2	65°	3	0·1273
0·1270	1	100	,, = 0·01 A.	3	65°	2	0·1268
0·1026	1	100	,, = 0·04 A.	2·5	65°	5	0·1026
0·1026	1	100	,, = 0·04 A.	2·5	65°	5	0·1024

TABLE III.

Silver. Grm.	Platinum. Grm.	Potassium cyanide. Grm.	Current.	Voltage.	Temperature.	Dilution. C.c.	Time. Hours.	Found. Grm.
0·1026	0·879	0·5	$N.D._{100}$ = 0·05 A.	2·5	78°	125	5	0·1030
0·1026	0·1758	1·25	,, = 0·05 A.	2·5	75°	125	3	0·1022
0·1026	0·1758	1·25	,, = 0·45 A.	2·4	75°	125	3	0·1028
0·1026	0·1758	1·25	,, = 0·04 A.	2·5	75°	125	3	0·1025
0·1026	0·1758	1·25	,, = 0·04 A.	2·5	75°	125	3	0·1025

TABLE IV.

Silver. Grm.	Copper. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1024	0·0998	2	125	$N.D._{100}$ = 0·03 A.	1·16	Cold	10	0·1027
0·1024	0·1	2	125	,, = 0·05 A.	1·1	65°	7	0·1026
0·1024	0·1	1	125	,, = 0·058 A.	1·66	65°	3	0·1026

TABLE V.

Silver. Grm.	Cadmium. Grm.	Potassium cyanide. Grms.	Dilution. C.c	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1024	0·168	2	125	$N.D._{100}$ = 0·02 A.	2·15	75°	4	0·1025
0·1024	0·168	2	125	,, = 0·02 A.	2·1	60°	4	0·1020
0·1024	0·168	2	125	,, = 0·025 A.	2·15	65°	5	0·1027

TABLE VI.

Silver. Grm.	Zinc. Grm.	Potassium cyanide. Grm.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1024	0·1	1	100	$N.D._{100}$ = 0·33 A.	2·76	60°	3	0·1026
0·1024	0·1	1	100	,, = 0·38 A.	2·73	70°	3	0·1028
0·1024	0·1	1	100	,, = 0·38 A.	2·70	65°	3	0·1027

TABLE VII.

Silver. Grm.	Nickel. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1024	0·1	1·5	125	$N.D._{100}$ = 0·02 A.	1·7	65°	3	0·1026
0·1024	0·1	1·5	125	,, = 0·03 A.	2·	65°	5	0·1025
0·1024	0·1	1·5	125	,, = 0·03 A.	1·66	60°	3	0·1025

TABLE VIII.

Silver. Grm.	Cobalt. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1024	0·1	2·75	125	$N.D._{100}$ = 0·038 A.	2·2	65°	5	0·1028
0·1024	0·1	3·5	125	,, = 0·02 A.	2·2	65°	3½	0·1027

TABLE IX.

Silver. Grm.	Iron. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1002	0·1	3	125	$N.D._{100}$ = 0·025 A.	1·8	65°	4	0·1001
0·1002	0·1	2·5	125	,, = 0·02 A.	1·8	70°	4	0·1005
0·1002	0·1	3·25	125	,, = 0·038 A.	2·2	65°	3	0·1000
0·1174	0·1	3	125	,, = 0·04 A.	2·25	70°	3	0·1176
0·1174	0·2	4	125	,, = 0·04 A.	2·2	70°	3	0·1174
0·1174	0·1	3	125	,, = 0·05 A.	2·2	75°	3	0·1175

I found that the following conditions gave accurate results. The solution of silver nitrate used contained 0·1024 grm. of metal and that of copper nitrate contained 0·0998 grm. of copper. To the mixed solution 2 grms. of potassium cyanide were added. The liquid containing an excess of potassium cyanide was diluted to 125 c.c. This was subjected to a current of $N.D._{100}=0·03$—0·05 A and V = 1·1—1·66. The current acted from three to seven hours. The deposit of silver was free from copper.

The results varied from the theoretical by 0·0002—0·0003 grm. (see Table IV.).

Silver from Cadmium.—Smith and Spencer (*Journ. Am. Chem. Soc.*, xvi., 420) found that the deposition of silver was accelerated by heating the solution during the electrolysis, and that while cadmium may be entirely precipitated in the cold by the current, yet with a low current and a temperature of 60° the cadmium is held in solution. A separation of silver and mercury may thus be effected in a cyanide solution. In the same journal, Smith and Frankel record the separation of mercury from cadmium, zinc, nickel, and cobalt. In 1895 Smith and Wallace published later results with the same method. Following the data thus obtained, a solution of silver nitrate containing 0·1024 grm. of silver and cadmium sulphate solution equivalent to 0·168 grm. of cadmium was used. The salts were transformed into their double cyanides by an excess of potassium cyanide. The silver was obtained free from cadmium in four hours. The current used was $N.D._{100}=0·02$ A and V = 2·15. The temperature was 60° before the current was passed through the solution (see Table V.).

The silver in all cases was found free from cadmium.

Silver from Zinc.—These metals were separated according to the following conditions:—Using zinc sulphate containing one-tenth grm. of zinc, and silver nitrate solution containing 0·1024 grm. of silver. The potassium cyanide used amounted to 1 grm. The solution was 125 c.c. in volume. It was electrolysed at 70° by a current of $N.D._{100}=0·032$—0·038 A and V = 2·76. In three hours the deposition of the silver was complete (see Table VI.).

Nickel and cobalt were separated from silver in a similar way.

Silver from Nickel.—A solution of nickel nitrate containing one-tenth grm. of nickel was added to a silver nitrate solution, and both converted into the double cyanides by $1\frac{4}{10}$ grms. of potassium cyanide. The solution was electrolysed with a current $N.D._{100}=0·02$—0·03 A and V = 1·7—2 for three hours. The separation in this time was found to be complete. The results show a variation from the calculated amount of +0·0001 grm. to −0·0002 grm. The solution of silver in nitric acid, when examined qualitatively, gave no evidence of the presence of nickel. The nickel solution, when examined, showed that the silver had been entirely precipitated (see Table VII.).

Silver from Cobalt.—Cobalt nitrate was the salt used in this separation. To the solution of silver nitrate (0·1024 grm. of silver) was added a solution of the cobalt salt containing 0·1 grm. of cobalt and 2·75 grms. of potassium cyanide. A current of $N.D._{100}=0·02$ A and V = 2·2—2·7 at a temperature of 65° was passed through the solution for five hours. The silver in this time was entirely precipitated. No cobalt was found deposited with the silver (see Table VIII.).

Silver from Iron.—Heretofore these metals have not been separated electrolytically in cyanide solution. A solution of ferrous ammonium sulphate, equivalent to one-tenth grm. of metallic iron, was treated with sulphurous acid in order to reduce any ferric iron which might exist. After the reduction was complete, the excess of acid was nearly neutralised with potassium carbonate; to this, $2\frac{4}{10}$ grms. of potassium cyanide were added. The solution of the double cyanide, dark green in colour, was added to a solution of silver which had been converted into the double cyanide by five-tenths grm. of potassium cyanide. The entire solution was made up to 100 c.c. It was heated to 65°, and electrolysed with a current of $N.D._{100}=0·04$ A and V = 2·7. At the expiration of three hours the silver was found to be entirely deposited. There was in some cases a separation of ferric oxide at the anode. The silver, after its solution in nitric acid, was tested for iron by ammonium thiocyanate. A faint pink colour, due to a trace of iron in the acid used for the solution of the metal, was observed. The deposit, which was light grey in colour, varied but slightly from the theoretical amount of the silver present in the solution (see Table IX.).

(To be continued).

ON THE PROGRESS OF CHEMISTRY IN GREAT BRITAIN AND IRELAND DURING THE NINETEENTH CENTURY.*

Part I.—From the Beginning of the Century to the Foundation of the Chemical Society (1841).

By T. E. THORPE, LL.D., F.R.S.,
President of the Chemical Society.

Although Century-summaries run a risk of being regarded as flat, stale, and unprofitable, I have ventured to think that it would not be uninteresting or altogether unuseful if, at this time, I sought to indicate in the broadest possible outline, what our countrymen have done during the past hundred years in gaining and extending knowledge within the special province of intellectual activity with which we, as a Society, primarily concern ourselves.

In examining the century's accounts, we naturally inquire into the nature of the balance with which it started. How much better it was felt to be than at the corresponding period of the eighteenth century may be gleaned from the circumstance that the Council of the Royal Society—actually the contemporaries of Newton—entered upon 1701 with the complaint, more petulant than dignified, that "the discouraging neglect of the great, the impetuous contradiction of the ignorant, and the reproaches of the unreasonable, had unhappily thwarted them in their design to perpetuate a succession of useful inventions." During the years that followed, the achievements of Newton's successors, Franklin, Dollond, Maskelyne, Canton, Priestley, Hunter, made the Fellows feel that they were not wholly dependent upon the patronage of the great, although as the battle over the lightning conductors too plainly showed, they were not altogether unmindful of the frowns and reproaches of the ignorant and unreasonable.

Let us then try to realise what was the state of things, so far as it may be thought to affect Chemistry and its pursuit, in the year 1800. To begin with, the population of these isles was less than half of what it now is. London contained only about 800,000 persons, and I need hardly say that the whole social and economic conditions of the people were vastly different from what they are to-day. In one respect, at least, the circumstances of this and that time were alike—we were at war. But the condition of the nation now is halcyon compared with what it was in 1800. England was then in such straits as she never was before. Her very existence as a nation was at stake. The harvest over a great part of the country had failed. Corn was upwards of £9 a quarter, and famine and pestilence brooded over the land. Whilst Napoleon's attempt at invasion had roused the nation to a defiant and stubborn resistance, the governing classes were torn asunder by party faction, and seemingly more eager to secure political supremacy than to devise means to resist the common enemy.

Under such untoward conditions, it might well be supposed that science would languish and decay. Such, however, was not the case. It is a singular fact, and the circumstance is not peculiar to our own history, that it is precisely during the lean years—during periods of national stress and strain—that some of our greatest intellectual

* Presidential Address o the Chemical Society, 1900.

triumphs have been won. In the history of Science, and especially of Chemistry, France was never more glorious than during the turmoil which preceded and followed the Reign of Terror; and in our own case never were such chemical triumphs achieved as during the gloomy period covering the dying years of the last century and the opening years of that which is now drawing to its close.

What then was the condition of chemical science in Great Britain in the year 1800? Who were its cultivators and what were they doing? Black had died in the preceding November; Priestley, who had suffered "the slings and arrows of outrageous fortune" in vainly buffeting with the times, had been settled some six years on the banks of the Susquehanna, occupying himself with his beloved theology and in futile efforts to resuscitate the dying doctrine of phlogiston. For, in 1800, phlogistonism had only been scotched in this country. Mr. Richard Kirwan, President of the Royal Irish Academy and Inspector-General of His Majesty's Mines in the Kingdom of Ireland, had, it is true, some years previously tried conclusions with the French chemists in his "Essay on Phlogiston," which had been translated by Mme. Lavoisier, in order to be refuted, point by point, by Lavoisier, Morveau, Laplace, Monge, Berthollet, and Fourcroy. The new theory was, in fact, indebted to this opposition for some of the strongest proofs on which it is founded. The nimble-witted Irishman found he was no match for this formidable battalion. He confessed himself vanquished, and, as he wrote to Berthollet, laid down his arms. Rather, he might have said, he went over to the enemy, for during the few years of activity which remained to him he preached the new doctrine with all the zeal of the convert, albeit a little restive at times under the tyranny of the nomenclature it imposed upon the chemical world. National prejudice, and the authority of Priestley and Cavendish, no doubt retarded its general adoption in this country. Cavendish, at the close of the last century, was in his 69th year; he had ceased to engage in chemical inquiry, although still keenly interested in its progress. James Watt, who played so notable a part in the events which led to the discovery of the composition of water, and younger by some five years than his quondam rival, was no longer actively occupied with chemical speculation. Wollaston, as dispassionate and hardly less reserved than Cavendish, was in his 34th year, struggling to get together the medical practice which his singularly cold and taciturn manner forbade him to expect. John Dalton, also in his 34th year, had just resigned his post as tutor at the Manchester Academy, where, as the Trustees were pleased to report, "he had uniformly acquitted himself to their entire satisfaction in the province of mathematics, natural philosophy, and chemistry," on the spacious salary of £80 a year, and was now looking out for pupils in those branches of learning at the rate of two shillings a lesson. Thomas Thomson was 27; Andrew Ure, of dictionary fame, was 22, the same age as Humphry Davy, who was still at the Bristol Pneumatic Institute, where he had published his "Researches, Chemical and Philosophical, chiefly concerning Nitrous Oxide." William Thomas Brande was a lad of 12; and little Michael Faraday, a child of 8, who lived in a mews near Manchester Square, keeping body and soul together on the weekly loaf he got from public charity, and whiling away his leisure in playing marbles in Spanish Place.

Of course the results of the more important chemical investigations of the period were laid before the Royal Society, and were published in the *Philosophical Transactions*, and subsequently in *Nicholson's Journal* or *Tilloch's Philosophical Magazine*. The Society at that time was, and had been for the previous twenty-two years, under the benevolent despotism of Sir Joseph Banks. The other officers were Wegg, Planta, Gray, and Layard. The meetings were held in the evening at 8 o'clock, and, of course, for the most part by candle-light, in one of the large rooms in Somerset House, or Somerset Place as it was then termed. The Society had moved, in 1780, from Crane Court, into apartments provided for them by the munificence of their patron, George III., and the skill of his architect, Sir William Chambers, which habitation, in the language of the Council, "it was hoped would confer on them an external splendour in some measure proportional to the consideration in which they had been held for more than a century." Hard by was the Crown and Anchor Tavern, where the Royal Society Club held its pious orgies; the expense of the dinner was limited to five shillings, and black puddings were a standing dish. It is evident that the drowsy philosophers did no violence to their digestion by a too prolonged attention to the intellectual fare of the evening, for even a comparatively short communication not unfrequently occupied two if not three sittings. The President, when in town, lived in the then fashionable neighbourhood of Soho Square. Cavendish's town house was at the corner of Montague Place and Gower Street, and from here he might be descried, in a faded violet court dress, frilled shirt front and wrists, with a great-coat of greyish-green, and a three-cornered cocked hat over his knocker-tailed periwig, slouching along, one hand behind his back, on the way to his library in Dean Street, Soho. Wollaston lived in Hunter Street, Brunswick Square, and subsequently in Dorset Street, Manchester Square; Smithson Tennant oscillated between his farm at Cheddar and his chambers in the Temple; and Charles Hatchett resided in the rural district of Hammersmith. Other notable chemical workers at this period were William Henry, of Manchester, William Haseldine Pepys, and Richard Chenevix.

There were, of course, other publishing agencies at work, with aims similar to those of the Royal Society, such as the Royal Society of Edinburgh, which grew out of a Society founded in 1731 by the leading medical men of that city; and the Royal Irish Academy, founded in 1782, and of which Kirwan was President for some years. Many of the provincial towns, too, had their Literary and Philosophical Societies, but with the exception of that at Manchester, to which the Henrys and Dalton were frequent contributors, their Proceedings and Transactions contain few important chemical memoirs during the first years of the century.

Theoretical chemistry was taught in the Universities, and in some of them, following the example set by Black, by means of experimental courses of lectures. At Oxford there had been Beddoes, but his political opinions were distasteful to the University, and he had resigned. At Cambridge there were F. J. Wollaston and Farish. Hope, who first made known the existence of strontia, lectured at Edinburgh; Robert Cleghorn at Glasgow; and Dr. French, who is credited with the saying that "Humphry Davy was a verra troublesome person in chemistry," taught at Aberdeen. Moreover, almost every large town had one or more teachers—men of the type of Varley, Garnett, and Aikin—who gave lessons at their private houses. Faraday relates how, during his bookbinding days, his master allowed him to go occasionally of an evening to hear the lectures delivered by Mr. Tatum, at his house in Dorset Street, of which he obtained knowledge by bills in the streets and shop-windows. The hour was 8 o'clock in the evening, and the charge was one shilling. More ambitious or more self-important people than Mr. Tatum announced their prospective courses among the *Miscellanea* of *Tilloch's* or *Nicholson's Magazines*. Thus, Dr. Beddoes informs the world that his "Lectures are to be calculated for both sexes and different ages; and that there may be little chance of exclusion by reason of narrow circumstances, the subscription is fixed at one guinea; but unless fifty persons shall have entered their names by the 31st of March, the Lectures will not go on, as without a tolerably numerous audience Dr. B. thinks he could bestow his time in a manner more advantageous to the public."

The principal manuals of the time were Kerr's translation of Lavoisier's "Elements"; Chaptal's "Elements,"

the standard text-book on applied chemistry; Parkinson's "Chemical Pocket-book or Memoranda Chemica"; Heron's "Elements"; Brisson's "Physical Principles of Chemistry"; Accum's "System of Theoretical and Practical Chemistry"; and Marcet's "Conversations."

The first two decades of this century are unquestionably the most momentous and the most brilliant of any period in the history of chemistry in our country. They witnessed the establishment of the fundamental laws of chemical combination and the atomic theory; the discovery of the so-called gaseous laws and of the real nature of the atmosphere; the application of voltaic electricity as an analytic agent; the isolation of the metals of the alkalis and of the alkaline earths; the determination of the chemical nature of the halogens; and in the discovery of tantalum, palladium, iridium, osmium, and rhodium, a considerable addition to the list of the metallic elements. These, in 1802, were only 23 in number, as against about 60 at the present time.

A number of new compounds were brought to light or identified during the same period,—such as fulminating mercury and fulminating silver, carbonic oxide, acetylene, phosgene gas,—for the most part substances deemed at the time of their discovery to be merely chemical curiosities, incapable from their very nature of being turned to useful account, but all of which are now of important practical application. The same period witnessed the invention of the reflecting goniometer and of the miner's safety lamp. It saw the establishment of gas-lighting, and with it the creation of an industry which has exercised a profound effect on the development of the chemical arts. It saw, too, the first attempt to control, by the aid of chemical analysis, the hygienic character of the drinking water supplied to a large community. The statement, in 1808, of Dr. Andrew Ure, who became Professor of Chemistry in Anderson's College in 1818, that the water from the wells in Glasgow, which were then the chief sources of supply, contained "a surprising quantity of heterogeneous matters in solution," paved the way for the action of Telford and Robertson Buchanan in bringing in "the inexhaustible supply from the River Clyde by means of pipes and steam-engines." In the *Philosophical Magazine* for 1808 we read that this was the first occasion on which the whole of a public supply was filtered "by means of reservoirs constructed for the purpose. . . . . This salutary process is effected by making the water filter through sand and gravel from the large reservoir into which it is first elevated by the steam-engine into a second reservoir posited a little lower, and from which the conveying-pipes receive their supply." It is not often that Glasgow is willing to take a lesson from Paisley, but it is interesting to read that a Paisley "buddy" first practised the filtration of water intended for public supply, and it is satisfactory to learn further that "this public spirited adventurer was amply remunerated for his expenditure."

(To be continued).

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th instant, The Duke of Norhumberland, K.G., President, in the Chair. The following were elected members:—Mrs. E. S. Beale, E. Callard, E. J. Duveen, E. Pearson, Lord Russell of Killowen, and J. A. Thierry. The special thanks of the Members were returned to Professor F. Clowes for his donation of £20 to the fund for the Promotion of Experimental Research at Low Temperatures. The Chairman announced that he had nominated the following Vice-Presidents for the ensuing year:—Sir F. Bramwell, Right Hon. Lord Lister, Dr. Ludwig Mond, Sir Andrew Noble, Mr. A. Siemens, the Hon. Sir J. Stirling, Sir J. Crichton Browne, and Sir William Crookes.

PROCEEDINGS OF SOCIETIES.

ROYAL SOCIETY.

A CONVERSAZIONE was held at the Society's Rooms, Burlington House, on Wednesday evening last, May 9th, the Fellows and guests being received by the President, Lord Lister, and the members of the Council.

Among the numerous interesting exhibits may be mentioned Messrs. H. B. Hartley and H. L. Bowman's demonstration of the properties of crystals yielding doubly-refracting liquids on fusion.

Certain crystalline organic compounds, viz., *p*-azoxyanisol, *p*-azoxyphenetol, and cholesteryl benzoate, have been found by Prof. Lehmann, of Carlsruhe, to give on melting (at temperatures of 116°, 134°, and 145° respectively) liquids possessing the properties of double-refraction and dichroism, even under conditions in which a state of strain is impossible. When these anisotropic liquids are further heated, they change at definite temperatures of transition (134°, 165°, and 178° respectively) into ordinary isotropic liquids. The intermediate bodies have been called "liquid crystals," for, although the evidence of their elasticity, viscosity, and dielectric capacity shows them to be undoubtedly liquids, yet nevertheless they possess, like crystals, both double-refraction and dichroism.

Another exhibit, which shows the possibility of directing the movements of torpedo boats, &c., at a distance, is shown by Mr. Richard Kerr, who has a clock to which a coherer is attached: a transmitter, in another part of the room, gives rise to ethereal waves on the Hertzian system; these acting on the coherer set the clock in motion.

Dr. Downing has some interesting maps showing the path of the total eclipse of the sun on May 28th next: this eclipse will be partially visible in London, viz., three-fourths of totality.

Mr. Thomas Thorp shows some celloidin grating films and their application to diffraction colour photography. Celloidin films of diffraction gratings, of about 14,500 lines to the inch, are impressed on chromated gelatin plates, the surface of which thus becomes grooved. Light falls on the gelatin surface through photographic transparency. The celloidin film is detached, and the plate immersed in warm water, when the image appears in grooves more or less decided. Pictures are superposed and illuminated, and are then seen in their natural or other desired colours stereoscopically.

Flashes induced by Hertz waves in a helium tube were shown by Prof. Minchin, and a number of examples of leadless glazed ware by Dr. T. E. Thorpe.

Some very good specimens of apparatus constructed of vitreous silica were shown by Messrs. W. A. Shenstone and H. G. Lacell. These specimens are a distinct advance on those exhibited last year.

A very delicate electric micrometer is exhibited by Mr. P. E. Shaw, by means of which movements as small as 1/200th of a wave-length of sodium light have been measured.

A variety of explosives, a few experiments, and the results of others, with models showing the effect of erosion in guns, were shown in the Meeting-room by Sir Andrew Noble at 9.45 o'clock.

At 10.30, Dr. A. W. Rowe gave a short demonstration of the photo-micrography of chalk fossils by reflected light; and at 11.15 Mr. F. Enoch showed on the screen a series of photographs showing the metamorphoses of one individual nymph of *Æschna cyanea*, from the moment of quitting the water to that of the perfect imago.

NOTICES OF BOOKS.

Practical Chemistry. Part I. By William French, M.A. (Cantab), F.I.C., F.C.S. London: Methuen and Co. 1900. Pp. 136.

After having gone through a preliminary course of laboratory work, such as weighing, thermometry, the use of burettes, and other apparatus, cork-boring, &c., the student should be able to go through the course of experimental chemistry as herein described without any difficulty or necessity for very minute instructions. The author's statement that his "object has been throughout the book to give the students as little information about the experiments as possible," is badly expressed; he gives ample explanations and directions for carrying out the experiments successfully, but does not overburden his instructions with unnecessary details. It should be remembered that most students take up the subject of chemistry voluntarily, and should be able to make observations and deductions from simple experiments, such as we here find, without much help.

The contents of this book are decidedly elementary, and go little beyond the simplest practical work. The subjects dealt with are mainly evaporation and distillation, filtration, and crystallisation, the constituents of air and water, earthy bodies such as chalk and lime, experiments with common soda, salt, and such like.

The appendices comprise a few details of fitting up apparatus, and a number of questions and exercises.

Aids to Practical Pharmacy for Medical Students. By A. Campbell Stark. Revised according to Pharmacopœia of 1898. London: Baillière, Tindall, and Cox. 1900. Pp. 170.

Small as this book is, it contains all the information necessary for a student who offers himself for examination in practical pharmacy of the Conjoint Board. After a few general remarks on pharmaceutical processes, medicinal preparations, prescribing, &c., a list of the substances used in pharmacy is given; this is divided into Part I., Inorganic Substances, and Part II., Organic and Vegetable Substances, in which their production, principal characteristics, preparation, and doses are described. In Part II. the drugs are arranged in groups, those drugs with similar pharmacological properties being placed together.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 15, April 9, 1900.

Density and Analysis of Sulphur Perfluoride.—H. Moissan and P. Lebeau.—By the action of excess of fluorine on sulphur the authors obtained a new gaseous body (sulphur perfluoride) having a formula SF_6. This gas is one of the heaviest known, its density being 5·03. The composition of this substance proves the hexatomicity of sulphur in a simple manner; it corresponds to sulphuric anhydride, SO_3. Its examination is very interesting, owing to the inertness of its properties. Two such active bodies as sulphur and fluorine, when united to saturation, give a compound which is not attacked by melted potash or by sodium at its fusion point. This new sulphur fluoride is totally different from the known chlorides of sulphur.

Heat of Formation of Hydrated and Anhydrous Strontium Dioxide.—M. de Forcrand.—A comparison of numbers obtained by the author shows that the heat of peroxidation of anhydrous SrO is inferior by 1 cal. to that of BaO. This explains the fact that anhydrous strontium dioxide is less stable and less easy to obtain than the protoxide. Its dissociation tension is greater. These numbers also show that this is the inverse of the precipitated hydrated state. Strontium dioxide has a greater affinity for water by 2 cal. than barium dioxide. As a result of this, hydrogen peroxide acts on dissolved strontia, evolving nearly as much heat as a strong acid, and giving a precipitate, $SrO_2.9H_2O$, which is the hydrated neutral salt.

A New Method of Fractionation of some Rare Earths. — Eug. Demarçay. — The author, in 1896, announced that samarium, of atomic weight 150, contained another element characterised by spectrum lines in the violet and ultra-violet. Since then he has been occupied in the isolation of this element. All known methods of fractionation apparently failed to isolate it, but a separation can be effected by the crystallisation from nitric acid of the double nitrate of magnesium and the rare earths. This double salt corresponds to the general formula $M_2(NO_3)_6.3Mg(NO_3)_2.24H_2O$. The lanthanum salt is the least soluble in nitric acid. Large quantities of pure samarium can be obtained by this method, whilst the new element is always associated with gadolinium, and so separated from samarium.

Formation of Monomercurammonium Iodide by the Action of Concentrated Ammonia on Mercurdiammonium Iodide. — Maurice François. — Concentrated ammonia, when added in large excess to mercurdiammonium iodide, allows of the formation of monomercurammonium iodide, $HgH_2.NI$, a body which has not previously been described. This iodide must be dried in a strongly ammoniacal atmosphere, and then extracted with pure ether, to eliminate the mercurdiammonium iodide. It is a white substance formed of microscopic crystals, differing from mercurdiammonium iodide in that it does not redden in the air and is not soluble in ether.

Crystalline Selenide of Manganese and an Oxyselenide.—M. Fonzes-Diacon.—(1) The author obtained the cubic manganese selenide, MnSe, (*a*) by the action of SeH_2 on a solution of manganese acetate slightly acidulated with HCl, (*b*) by the reduction of selenate of manganese in the electric furnace by means of carbon, and (*c*) by the fusion of precipitated manganese selenide at a high temperature. (2) He obtained a prismatic manganese selenide, MnSe, by the action of SeH_2 on manganese chloride at a red heat. (3) By reducing manganese selenate by means of hydrogen at a high temperature, green oxyselenide of manganese was prepared.

Reducing Action of Calcium Carbide.—M. Geelmuyden.— Moissan has previously noticed the reducing properties of calcium carbide, and the reactions which take place in the presence of oxides. The author examines the action of calcium carbide on various other substances in the electric furnace. The commercial material was used for these experiments, and its action on boric anhydride and several metallic sulphides examined.

New Method of Production of Double Sulphates of Chromium.—C. Pagel.—When sulphuric acid acts on bichromate of potash and potassium chloride, only hexagonal crystals are obtained. When, however, bichromate of soda and pure sodium chloride are used, a prismatic salt is obtained. These crystals are insoluble in water and acids, and are not attacked by soda solution in a sealed tube. When heated on platinum foil they become reddish violet, and on cooling re-take their green colour. Analysis shows them to have the composition Cr_2O_3, 18·5; SO_3, 58·62; $SO_4.Na_2$, 52·24. Numbers which correspond to the formula $(SO_4)_3Cr_2.3SO_4.Na_2$.

Electrolytic Estimation of Lead in Sulphates and Chromates. Application to the Analysis of Lead Glass and Lead Chromates.—C. Marie.—The lead sulphate is placed in a glass electrolytic cell, then attacked by nitric acid, crystals of ammonium nitrate being added little by little. When the sulphate has completely disappeared, the whole is diluted with hot water, and electrolysed under ordinary conditions, keeping the temperature at 60—70°. The lead dioxide is weighed on the electrode. This method can be applied to the analysis of lead glass, by adding HF to the finely-powdered glass, and a sufficient quantity of sulphuric acid to transform the bases into sulphates. Chromate of lead dissolves in nitric acid, and ammonium nitrate still more easily than the sulphate.

α-α-β-Trimethyl-β-oxyadipic Acid.—E. E. Blaise.—The methylic ether of the lactonic acid corresponding to the ααβ-trimethyl-β-oxyadipic acid, has been obtained by condensing methyl levulate and methyl bromoisobutyrate in presence of zinc. The bromozincic derivative thus formed is then decomposed by dilute sulphuric acid. This condensation only gives a yield of about 15 to 17 per cent.

Action of Amyl Chloride on Calcium Carbide.—P. Lefebvre.—By passing the vapour of amyl chloride over calcium carbide heated to redness various products are obtained. The liquid products, when fractionally distilled, separate into products not containing chlorine boiling below 45°, and products containing chlorine boiling above 75°. These products were analysed and identified as far as possible. The analysis of the gases obtained showed the presence of acetylene, ethylene, other ethylenic carbides, hydrogen, and nitrogen.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 1.

Normal Presence and the Localisation of Arsenic in the Organs of Animals.—A. Gautier.—Already inserted (p. 135).

Malates and Tartromalates.—Ch. Ordonneau.—Tartromalate of lime α is prepared in the pure state by treating crude tartrate of lime with boiling water. The filtered liquid, after cooling for twelve hours, gives 5 or 6 grms. per litre. Under the microscope it appears in long silky needles. When air-dried, it contains 9 to 10 H_2O. 100 c.c. of distilled water at 20° dissolved 0·27 grm. of this salt, but it is much more soluble in boiling water, by which it is, however, partially decomposed. The same salt is obtained by mixing equal weights of tartaric and malic acids, saturating with ammonia, and precipitating the solution with excess of acetate of lime, added all at once. The author also describes the preparation and properties of malate of lime α, tartromalic acid α, bimalate of lime, malate of lime β, tartromalate of lime β, and tartromalic acid β.

Some Chloranisidines and *m*-Chloranisol.—F. Reverdin and F. Eckhard.—To decide the question as to whether or no there was a migration of the halogen in the nitration of *p*-chloranisol, the authors took up the comparative examination of the nitration of the ortho- and para-chloranisols, and they made use of the opportunity to examine the characteristics of the derived chloranisidines. The three substances are easily recognised by means of their nitrated derivatives; the *o*-chloranisol, on nitration, gives *o*-chlor-*p*-nitranisol, fusible at 95°, not distilling with steam; *p*-chloranisol gives *p*-chlor-*o*-nitranisol, fusible at 98°, distilling with steam; finally, under the same conditions, *m*-chloranisol gives a derivative fusible at 58°, distilling with steam.

On Dissymmetric Tetramethyldiamidodiphenylethane.—A. Trillat.—This body occurs as a white mass, perfectly crystallised, in the form of flakes fusing at 68—69°; it is insoluble in water, but soluble in alcohol, ether, ligroïn, chloroform, and all dilute acids, with which it forms well crystallised salts. When exposed to the air it takes a red tinge, probably due to some intra-molecular transposition.

Examination of some Derivatives of Leucobase, $C_{18}H_{24}N_2$.—A. Trillat.—If leucobase is dissolved in acetic acid, and a little binoxide of lead be added, a beautiful greenish-blue colour immediately appears, and carbinol is formed. This organo-plumbic compound is decomposed by soda after two hours, the hydrate of lead being dissolved and the base left. This treatment is repeated several times, and the base then washed with boiling water; when dry, it appears as a brown mass, soluble in all acids, but particularly so in acetic acid, taking a blue colour. The author then describes a number of salts, such as the chloroplatinate, the acid sulphate, the acetate, phosphate, oxalate, bromethylate, &c., and the nitrated, bromised, and iodised derivatives.

Action of Nitrous Acid on Leucobase, $C_{18}H_{24}N_2$.—A. Trillat.—Already noticed.

Action of Chloride of Aluminium on Camphoric Anhydride.—G. Blanc.—Already noticed.

Benzoylfurfurane.—R. Marquis.—Already noticed.

Action of Hydroxylamine and Phenylhydrazine on Dithiobenzoylacetone.—V. Vaillant.—Dithiobenzoylacetone, $\begin{matrix} C_6H_5—CO—CH—CO—CH_3 \\ \\ C_6H_5—CO—CH—CO—CH_3 \end{matrix} > S_2$, of which a certain number of reactions have already been described, combines easily with hydroxylamine and phenylhydrazine, giving the corresponding oxazol and pyrazol.

The Essences of Geranium.—MM. Jeancard and Satie.—The authors have examined essences of geranium from six different sources. The solubility of all of them in alcohol is practically the same; that is, 1 volume of essence dissolves in 0·9 to 1 vol. of alcohol at 80°, or in 2 to 2·3 vols. at 70 per cent at 15°. They contain variable quantities of acids, and become oxidised on contact with the air. That from Cannes contains the least free acid and oxidises the least rapidly.

Absorption of Iodine by Plants.—P. Bourcet.—Already inserted in full (p. 110).

On a Cause of Error in Testing for Sugar in Urine by means of Fehling's Solution.—J. Eury.—The presence of sugar in urine must not be assumed only when a precipitate of suboxide of copper is formed by the cupropotassic solution. When a urine containing a large amount of sugar is heated with cupro-potassic solution, a rapid decolouration is observed, while the liquid remains limpid. When left to itself, the upper part becomes rapidly of a reddish brown colour, and a brown precipitate is soon formed throughout the tube. This is due to an oxidation only produced by heat, but the precipitate is not suboxide of copper, and it must not therefore be concluded that sugar is present. The reaction is found to be due to the presence of creatinic bases.

Estimation of Phosphorus in Organic Compounds.—Ch. Marie.—Already noticed.

The Salts of Pyropervanadic Acid.—P. Melikoff and L. Pissarjevsky.—By treating the metavanadates with H_2O_2 we obtain the salts VO_4M. If we add an excess of alkali to the H_2O_2 we obtain the yellow compounds, $V_2O_{11}(NH_4)_4$ and $V_5O_{26}K_8.2H_2O$, which are considered by the authors to belong to pyropervanadic acid, the potassic salt being $3VO_4K_2O_2+2VO_4K+2H_2O$. By dissolving this latter salt in H_2O_2, adding KOH, cooling the mixture to 0°, gradually adding H_2O_2 until the green colour no longer changes, and precipitating with alcohol, we obtain a fluorescent precipitate having the formula $V_2O_9K_2.3\frac{1}{2}H_2O$, that is to say, a compound of pyropervanadic acid with K_2O_2. All these compounds are fairly stable in the dry state.—*Journ. Soc. Phys. Chim. R.*, xxxi., p. 108.

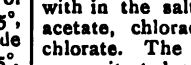
MISCELLANEOUS.

Volumetric Estimation of Bismuth by Means of Arsenious Acid in Alkaline Solution.—C. Reichard.—This method consists in transforming the bismuthic compound into Bi_2O_5 by means of a current of chlorine in alkaline solution. Wash by decantation to remove the Cl and NaOH. Add excess of a solution of As_2O_3 in potash or soda-lye, and boil until the red precipitate becomes white. Filter and estimate the remainding As_2O_3 with permanganate.—*Zeit. Anal. Ch.*, xxxviii., p. 100.

Digestion of Fibrine and Albumen by Papaine, and on a New Coloured Reaction of the Products obtained.—V. Harlay.—Three digestions were made with a papaine which dissolved fibrine rapidly: *a* was in a solution containing 0·2 per cent of HCl; *b* contained 0·2 per cent of bicarbonate of soda; and *c* was in simple aqueous solution. All three contained 0·10 grm. of papaine per 50 c.c., and were made to act on 5 grms. of fibrine. They were heated to 40—45° on the water-bath. The disaggregation was almost as rapid in *b* as in *c*, and almost total in two hours and a half. In *a*, there was first swelling and then solution, but even after six hours there still remained a considerable amount of fibrine, swollen but not dissolved. The liquid *a* was difficult to filter, but *b* and *c* filtered easily; the polarimeter showed that the action in *b* was less advanced than in *c*. All three liquids gave with tyrosinase the characteristic colouration—first red, then green,—but the two former had to be first neutralised. The colouring-matters produced by the action of tyrosinase on papaïc and pepsic liquids and digestions are identical; and, in the case of pepsic digestion, this colouring-matter is accompanied by other colouring-matters, which are the first destroyed by reduction with zinc and hydrochloric acid. This reaction distinguishes papaïc from pancreatic digestion, and separates it from pepsic digestion.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xi., No. 4.

Some Compounds of Iodantipyrine with the Mercuric Salts.—J. Bougault.—When 3·14 grms. of iodantipyrine, dissolved in 30 c.c. of warm alcohol at 95°, is mixed with a solution of 2·71 grms. of bichloride of mercury in 20 cubic centimetres of alcohol at 95°, there are deposited, on cooling, beautiful colourless crystals of $C_{11}H_{11}IN_2O.HgCl_2$. This compound melts at 168—169° with decomposition. It is almost insoluble in hot or cold water; it is slightly soluble in cold alcohol, and insoluble in ether. The bi-iodide of mercury does not combine directly with iodantipyrine under similar circumstances, though bi-iodide of mercury combines easily with antipyrine, forming $(C_{11}H_{11}IN_2O)_2.HgI_2$. The compound $(C_{11}H_{11}IN_2O)_4.HgCl_2.HgI_2.2HCl$, is formed by the action of iodine on antipyrine in the presence of an excess of sublimate in alcoholic solution; when the solutions are sufficiently concentrated, the crystals are deposited; they are white or pale yellow in appearance, and melt with decomposition at about 140°. This body is also decomposed very rapidly by water; the rose tint of the bi-iodide of mercury rapidly appears, and the hydrochloric acid goes into solution with a little of the mercuric salt. The compound $(C_{11}H_{11}IN_2O)_2HgI_2.HCl$ is obtained by the action of iodine on antipyrine in alcoholic solution in the presence of a definite quantity of sublimate (2 mols. of $HgCl_2$ for 4 mols. of $C_{11}H_{12}N_2O)_2$. This body occurs in crystals having a beautiful yellow appearance; it melts with decomposition at 130°; with regard to the action of solvents, it behaves exactly the same as the body $(C_{11}H_{11}IN_2O)_4HgI_2HgCl_2HCl$.—*Journ. de Pharm. et de Chimie*, Series 6, vol. xi., No. 4.

Tungsten.—A. Stavenhagen.—To prepare metallic tungsten the author took an intimate mixture of pure tungstic anhydride and aluminium filings, also as pure as possible (2 mols. Al to 1 mol. WO_3); 300 grms. of this mixture were placed in a clay crucible, and the fire started by means of a magnesium ribbon, ending in a cap formed of BaO_2, and Al filings. The reaction is almost explosive, and a part of the contents of the crucible are thrown out. After cooling, we find disseminated through the slag, a friable, porous, metallic mass consisting of tungsten containing 2·6 per cent of aluminium, not attacked by acids, even aqua regia, easily attacked by melted KOH. To obtain melted tungsten, the author, after having proceeded by deflagration as just described, added a certain quantity of sheet aluminium to the mass, and submitted the whole to the action of a large oxy-hydrogen flame; after fusion he continued to inject oxygen alone; the excess of aluminium was thus burnt off, and after cooling, a button of tungsten was found under the slag, not containing more than 0·5 per cent of aluminium, in a crystalline mass, in colour a little darker than zinc, and scratching glass; its density was 16·6; it is unattacked by acids, and slowly attacked by melted KOH. Tungsten only melts superficially in the Moissan furnace; it loses its aluminium, but has a tendency to become carburised. As it is a bad conductor of electricity, it does not directly receive the effect of the voltaic arc, which prefers going round by a path of less resistance.—*Berichte*, xxxii., p. 1513.

The Chemistry of Mercury.—H. Ley and H. Kissel.—The authors have examined the electric conductivity of the solutions of a certain number of mercuric salts, with a view to determining their constitution; they start with the principle that the hydrolysis of these salts in the cold is much more marked (in the same way as the electric conductivity of water is increased), if the mercury is united directly with the oxygen, than if it is in direct relation with another metalloid (N, C, Cl). This latter is the case with the chloride, bromide, and cyanide of mercury as well as the acetymide, succinamide, and even mercuric glycocol, which seems to show that in this latter case the glycocol has taken the form  . The first case (strong hydrolytic dissociation), on the other hand, is met with in the salts of oxygenated acids, such as mercuric acetate, chloracetate, propionate, glycolate, and perchlorate. The authors then pass to the examination of some nitrated salts. Mercuric nitrite may be compared with the acetate, the mercury is attached to the oxygen. The mercuric salt of methylnitramine (fine, hard needles, slightly soluble in water), is also easily dissociated in its solutions; methylnitramine must therefore be regarded as a hydroxylised body, $CH_3.N=NO.OH$. On the other hand, fulminate of mercury behaves like the cyanide, from the point of view of its decomposition by water and its electric conductivity; we know that these two salts react in a similar manner with regard to sodium amalgam, being transformed into a sodic salt. It is therefore probable that fulminic acid, C=N−OH, undergoes, in its mercuric salt, a tautomeric transformation which places H in connection with C, or with N, as happens with various oximes. Mercuric nitroform, $[C(NO_2)_3]_2Hg$, is obtained easily by agitating an ethereal solution of nitroform with HgO; it occurs in colourless prisms, extremely soluble in ether, giving a colourless solution; it behaves in the same way with benzene, chloroform, and acetic ether. Its solutions in alcohol, acetone, and acetaldoxime is of a pale-yellow colour; its solutions in water, pyridine, and benzonitrile are strongly yellow. With water this colour comes from dissociation; with pyridine from combination. The aqueous solutions are acid, and are dissociated very easily; the authors, agreeing with the views held by M. Hantsch (*Ber.*, xxxii., 580), think that the salt, either solid or dissolved in non-dissociating solvents, is a metallic derivative or pseudo-salt, with Hg near to C, while in an aqueous solution, there is a formation of free nitroform and of a derived basic salt, of a hydroxylised tautomerous form.—*Berichte*, xxxii., p. 1357.

The Salts of Perboric and Pertitanic Acids.—P. Melikoff and L. Pissarjevsky.—The authors have arrived at the following conclusions:—Perboric acid, BO_3H, with the alkaline metals forms soluble salts fairly stable in the dry state. The salts of the alkaline-earthy and the heavy metals are either only slightly soluble or else insoluble. The acid BO_3H with the peroxides only gives extremely unstable compounds. Besides the acid TiO_3, there is still probably an acid Ti_2O_7. Both of them easily form either basic or neutral compounds with the peroxides of the alkaline and alkaline-earthy metals. All these compounds are fairly stable.—*Journ. Soc. Phys. Chim. R.*, xxx., p. 693.

The Reduction of Benzile.—J. Thiele.—According to a previous paper, the reduction of benzile ought to give rise to a non-saturated glycol:—

$$C_6H_5.\overset{\displaystyle O}{\overset{\|}{C}}-\overset{\displaystyle O}{\overset{\|}{C}}.C_6H_5 + H_2 = C_6H_5\,\dot{C}{=}\dot{C}.C_6H_5$$

But this latter is unstable, and is immediately transformed into benzoïn. If the reduction is effected in the presence of acetic anhydride, the glycol can be isolated in the form of diacetic ether. This glycol is not a product of transformation of benzoin, as this latter gives an acetylised derivative directly when treated with acetic anhydride. The diacetic ether of diphenylglycol occurs in two stereoisomeric forms (ethylenic isomers). The reaction is produced in the following manner:—50 parts of benzile are dissolved in 300 parts of acetic anhydride; then, after cooling, a mixture of 50 c.c. of sulphuric acid and 100 c.c. of acetic acid are added. To the liquor, kept at 30 to 40°, 60 grms. of powdered zinc are gradually added. The solution is then precipitated with water, exhausted with ether, and crystallised in benzene. The less soluble modification α is first deposited in the form of white needles, fusible at 153°, slightly soluble in organic liquids; the more soluble isomer β crystallises in flakes, fusible at 110°. Alcoholic potash saponifies both the diacetates, giving benzoïn. Permanganate oxidises them easily, but bromine does not have much effect. By heating the isomer β for five hours with acetic anhydride it is transformed, to a very small extent, into the isomer α.—*Ann. Chem.*, cccvi., p. 142.

MEETINGS FOR THE WEEK.

Monday, 14th.—Society of Arts, 8. (Cantor Lectures). "The Incandescent Gas Mantle and its Use," by Prof. Vivian B. Lewes.

Tuesday, 15th.—Royal Institution, 3. "Brain-Tissue considered as the Apparatus of Thought," by Alex. Hill, M.A., M.D.

Wednesday, 16th.—Society of Arts, 8. "A National Repository for Science and Art," by Prof. Flinders Petrie.

—— Microscopical, 8. "On the Lag in Microscopic Vision," by E. M. Nelson, F.R.M.S. Preceded at 7.30 by an Exhibition of Microscopic Pond Life.

—— Institution of Mining and Metallurgy, 8. "Presidential Address," by C. Algernon Moreing (President). "The Mineral Resources of New Caledonia," by F. Danvers Power, M.I.M.M. "Notes on Gold and Silver Amalgamation," by W. S. Welton, M.I.M.M.

Thursday, 17th.—Royal Institution, 3. "A Century of Chemistry in the Royal Institution," by Prof. Dewar, M.A., LL.D., F.R.S.

—— Chemical, 8. "Chlorine Derivatives of Pyridine—VI. The Orientation of some Aminochloropyridines," by W. J. Sell, M.A., and F. W. Dootson, M.A.

—— Society of Arts, 4.30. "The Industrial Development of India," by Jervoise Athelstane Baines, C.S.I.

Friday, 18th.—Royal Institution, 9. "The Structure of Metals," by Prof. J. A. Ewing, M.A., F.R.S.

Saturday, 19th.—Royal Institution, 3. "South Africa—Past and Future," by Dr. Alfred Hillier.

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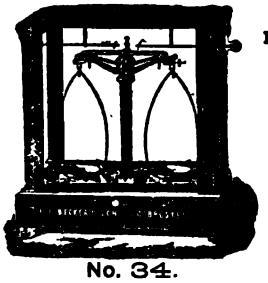

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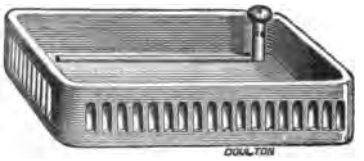

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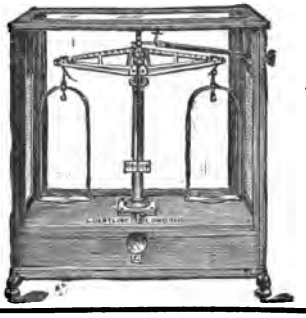

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Vol. LXXXI., No. 2112.

THE ACTION OF CHLORIDE OF CALCIUM ON SILICATES.

By A. TIXIER.

When a silicate is heated to redness with chloride of calcium, chlorine is given off if the calcination is effected in a current of air, and hydrochloric acid if we direct a current of steam on to the red-hot mixture. This reaction has even been the subject of many applications for Solvay patents since 1877, either with regard to the principle itself or for the methods of application. The Solvay patents particularly mention the use of silica, alumina, silicates of alumina, clays, &c., which, it is true, are the most common. They make no allusion to the more complex silicates, such as the felspars, lepidolites, petalites, &c. Further, there does not appear to have been published any work mentioning the nature of the reactions which occur during this method of treating chloride of calcium with the object of utilising the chlorine, nor on the influence of the composition of the mixture on the result. The present paper fills up these gaps, and will further show that secondary reactions take place which are capable of important commercial applications.

When we heat a silicate with chloride of calcium, if the silicate contains alkalis they are transformed into chlorides, which are easily removed by simply washing with water. If the calcination is effected in a current of air, chlorine is given off, mixed with a certain quantity of hydrochloric acid, coming either from the water of hydration of the silica or from the moisture remaining in the mixture.

The return of chlorine depends essentially on the composition of the silicate and its proportion with regard to the $CaCl_2$; the amount of chlorine liberated is so much the greater, under identical circumstances, as the silicate is richer in silica and poorer in alkalis and in aluminium. The maximum returns in chlorine were obtained with pure silica. By this means, as much as 96 per cent of the total chlorine was recovered, of which 90 per cent was in the form of free Cl_2 and 10 per cent in the state of HCl. When the object of the reaction is only to produce chlorine, it is thus not advantageous to use silicates of alumina. The proportion of silicate with regard to the chloride has also a great influence on the return. This increases with the proportion of silicate, but only up to a certain limit; after that an excess is a disadvantage; this limit is further reduced when, at the same time as the production of chlorine, we want to collect the alkali from the silicate. In both cases excess of silicate makes the mass, which has till then kept porous, more fusible, and thus diminishes the action of the air; it also reacts on the alkaline chlorides formed, giving insoluble complex chlorosilicates, from whence arise losses in chlorine and in alkali.

The experiments described in this paper were carried out with the following materials:—Pure alumina, lepidolite, kaolin, petalite, artificial silicate of alumina, and silicates from various sources. The experiments were made with the double object of obtaining maximum quantities of chlorine, and, in the case of silicates containing alkalis, especially lithia, of extracting these alkalis under the most favourable conditions.

The following was the composition of the materials used:—

Alumina.—The pure commercial product used in the manufacture of aluminium.

Lepidolite—

Loss at a red heat	4·15	
Silica	54·50	or 56·08 per cent of the anhydrous mineral.
Alumina	29·80	
Oxides of Fe and Mn	Traces	
Lime	Traces	
Potash	9·00	
Soda	Traces	
Lithia	2·80	
Fluorine	Traces	

Kaolin—

Loss at a red heat	13·00	
Silica	52·00	or 59·77 per cent of the anhydrous substance.
Alumina	36·05	or 40·23 per cent of the anhydrous substance.

Petalite—

Loss at a red heat	1·4	
Silica	74·50	
Alumina	20·75	
Oxide of iron	Traces	
Lime	3·00	
Lithia	2·30	

Artificial Silicate of Alumina—

Moisture	51·00	
Silica	39·00	or 79·05 per cent of the anhydrous substance.
Alumina	4·50	
Soda	5·50	

Silicas containing from 85 to 99 per cent of SiO_2 (differences = water, lime, alumina).

Experiments with regard to the Manufacture of Chlorine.—The apparatus consists of a porcelain tube heated over a row of burners and enclosed in an iron tube. A current of dry air is forced in at one end, the other end of the tube is connected with two flasks, followed by a vertical tube filled with glass beads. By a very simple arrangement the smell can be judged towards the end of the operation without disconnecting the apparatus, and with hardly any loss. The method of working is as follows:—A double normal solution of pure chloride of calcium is used, taking 50 c.c. for each operation; that is equal to 3·55 grms. of chlorine. This solution is evaporated to dryness with the corresponding silicate in a small porcelain crucible, while constantly stirring with a glass rod. The dry material is then detached and placed in the tube—either in a porcelain boat, or, if the volume is too great, in the tube itself. The crucible and the glass rod are both rinsed with distilled water, and the chlorine in solution estimated; thus, by difference, we get the quantity of chlorine placed in the tube. Heat to redness, and then pass dry air through at the rate of three bubbles a second. The first washing flask contains 50 c.c. of a double normal solution of arsenite of soda (1 litre = 71 Cl), which absorbs the Cl and HCl. The second flask and the vertical tube contain water tinted with litmus. After the operation, which lasts four hours, the Cl set free and the total Cl are estimated. The contents of the first wash-bottle are diluted to 500 c.c., and the remaining As_2O_3, and thus the Cl_2 absorbed, is titrated with N/5 iodine in this solution. The total chlorine is estimated by precipitation by means of NO_3Ag in nitric solution. The contents of the tube are further washed with distilled water; in this solution (filtered) the Cl is estimated; the insoluble residue is attacked with nitric acid, and the Cl again estimated in this fresh solution. In this manner the whole of the chlorine is accounted for, and the amount lost during the operation is estimated. The following is an example of the method of working (Experiment 20 in

the table):—Action of silica on chloride of calcium in the proportion of $2CaCl_2 : SiO_2$.

Dry together { 50 c.c. of $2NCaCl_2$ solution = 3·55 Cl. / 9·5 grms. anhydrous silica.

The product is detached, and there remains adhering to the crucible 0·054 Cl. Calcine for four hours in a current of air (3 bubbles a second); the residue, after calcination, weighs 5·96 grms.

The chlorine recovered is—

	Grms.	
In the drying crucible	0·054	
Given off as free Cl_2	1·003	Total, 1·263 grm.
Given off as free HCl	0·260	

Chlorine in the residue from calcination—

Soluble in water	1·769
Soluble in NO_3H	0·386
Total	3·472
Loss (3·55 − 3·472) . .	= 0·078

There is therefore given off—

$$\frac{1·263}{3·55 - 0·054} = 36·1 \text{ per cent of total Cl,}$$

containing—

$$\frac{1·003}{1·263} = 79·4 \text{ per cent of free chlorine.}$$

The experiments made with regard to the production of chlorine are given in the accompanying table. At the same time the return of lithia is given; this shows the influence of the excess of silicate. The figures given in this table are, above all, of relative value; for, of all the operations, which were made under identical conditions of heating and duration (four hours at a cherry-red heat), none was pushed to the extreme limit of exhaustion; the most important action, the liberation of chlorine, became slower and slower, though it never ceased entirely.

Experiment 26 was made under conditions which enabled an account to be kept of the variations in the liberation of the chlorine. The gases, on leaving the porcelain tube, passed through a drawn-out tube into a beaker containing a solution of iodide of potassium and starch. The chlorine as it passed over displaced the iodine, which coloured the solution blue. By the aid of a graduated burette, a titrated solution of arsenite of soda was added, so as to keep the solution colourless. In this way it was found that—

During the first hour . .	0·78	of free Cl came off.
„ second hour . .	0·54	„ „
„ third hour . .	0·556	„ „
„ fourth hour . .	0·487	„ „
Total	2·363	

And at the same time 0·080 of HCl

Experiments on the Treatment of Silicates.—The treatment of silicates containing alkalis by chloride of calcium enables us to effect the extraction of these alkalis under conditions of high return and economical working that cannot be attained by any other process at present known. It further enables us to treat silicates—such as petalite—which are only attacked with difficulty by the usual reagents, with great ease, and to effect a veritable commercial analysis. The proportions of chloride of calcium and silicate to be used vary according to the composition of the silicate; the manner and duration of the heating have also a great influence on the success of the operation; but these conditions are easily arranged after a few laboratory trials. In this manner we change the alkalis into chlorides, using only a slight excess of chloride of calcium. A methodical washing with water removes all the chlorides, and they are easily dealt with afterwards. The residue insoluble in water—formed of silicate of alumina and lime—is very easily attacked by HCl, with an abundant deposit of gelatinous silica. The magma thus obtained can be easily dried at 120°, owing to the $CaCl_2$ it contains, and gives an insoluble silica which remains, after being taken up with water acidulated with HCl, in the form of a dense easily washed sand. Finally, it suffices to evaporate the solution of $CaCl_2$ and Al_2Cl_6 obtained to dryness, and to heat the residue to 300° in a current of air to obtain, after washing with water, a very pure alumina, free from iron and lime, and very suitable for the manufacture of aluminium and its derivatives. The $CaCl_2$ can be used over again for the treatment of fresh quantities of silicates, or for making chlorine with the help of the very pure silica also obtained.

No.	Material and proportion.	Silica per cent of anhydrous silicate.	Total Cl given off.	Chlorine. Free.	Chlorine. As HCl.	Lithia obtained per cent of silicate.
1.	$CaCl_2 + Al_2O_3$ ..	0	7·37	88·2	11·8	—
	$CaCl_2$. Lepidolite.					
2.	1 : 1 ..	56·8	36·1	68·8	31·2	—
3.	1 : 1 ..	—	35·3	60	40	—
4.	1 : 2 ..	—	36·3	54·06	45·94	—
5.	1 : 2 ..	—	29	47	53	—
6.	1 : 2 ..	—	41·7	14·5	85·50	2·12
7.	1 : 2·57	—	47·3	38·6	61·40	1·45
8.	1 : 2 ..	—	38·6	—	—	2·10
9.	1 : 3 ..	—	50	43·8	56·2	0 92
10.	1 : 4 ..	—	60·2	35·25	64·75	0·77
11.	1 : 5 ..	—	58·1	30·29	69·71	0·46
	Moist kaolin.					
12.	1 : 1 ..	59·77	35·8	62·6	17·4	—
	Calcined kaolin.					
13.	1 : 1 ..	—	58·5	77·9	22·1	—
14.	1 : 2 ..	—	84·6	68·8	31·2	—
	Petalite.					
15.	1 : 1 ..	75	71	75·32	24·68	2·01
16.	1 : 2 ..	—	57·75	70·50	29·50	—
17.	1 : 2 ..	—	42·1	—	—	—
18.	1 : 3 ..	—	60·5	68·40	31·60	—
	Artificial anhydrous silicate of Al_2O_3.					
19.	1 : 1·2	75	71·6	76·9	23·1	—
20.	$2CaCl_2 + SiO_2$.	98	36·1	79·4	20·6	—
21.	$CaCl_2 + SiO_2$.	98	62·3	81·6	18·4	—
22.	$CaCl_2 + SiO_2$.	97	85	75·4	24·6	—
23.	$3CaCl_2 + 4SiO_2$.	98	88	72·1	27·9	—
24.	$3CaCl_2 + 4SiO_2$.	98	89·2	72·4	27·6	—
25.	$CaCl_2 + 2SiO_2$.	98	89	74·9	25·1	—
26.	$CaCl_2 + 2SiO_2$.	92	69·7	96·7	3·3	—
27.	$CaCl_2 + 3SiO_2$.	98	89·5	82·6	17·4	—
28.	$CaCl_2 + 3SiO_2$.	85	62·8	76	24	—
29.	$CaCl_2$ 1 : SiO_2 2	99	89·8	92·2	7·8	—
30.	$CaCl_2$ 1 : SiO_2 2	99	96·05	90·03	9·97	—

This method, applied to the treatment of lepidolite and petalite, which have already been used in the chlorine experiments, gave the following results, working on 100 grms.:—

Lepidolite.

Proportion: 2 Lepidolite to 1·5 Chloride of Calcium.
Time of Heating: Five hours at red heat.

	Obtained.	Theory.
Silica	53·00	54·5
Alumina	27·00	29·0
Chloride of potassium	14·14	14·20
Lithia	2·77	2·80

Or 97·1 per cent of the lithia present.

Petalite.

Proportion: 1 Petalite to 1 Chloride of Calcium.
Time of Heating: Five hours at red heat.

	Obtained.	Theory.
Silica	75·5	74·5
Alumina	17·0	20·75
Lithia..	2·30	2·30

Or 95·6 per cent of the lithia present.

Such returns are unobtainable by any other method in use up to the present time.—*Moniteur Scientifique*, Series 4, vol. xiv., April, 1900.

ON A GAS OBTAINED FROM CYANOGEN WHICH APPEARS TO BE IDENTICAL WITH ARGON.

By Dr. T. L. PHIPSON.

When dry cyanogen is passed into a glass tube heated to redness, and full of iron nails (*points de Paris*), devoid of rust, a gas is obtained which can be collected over water, in which liquid it is only slightly soluble. This gas is colourless, and almost odourless; it extinguishes a lighted match, without taking fire itself; it is not absorbed by potash, and when mixed with oxygen and submitted to the electric spark it does not detonate. 1000 c.c. weighed in two experiments 1·800 and 1·810 grms. at 0° C. and 30 inches barometer. These weighings were made with the best balance in my laboratory. This gas is therefore not nitrogen (of which 1000 c.c. in the same circumstances weigh 1·267 grms.), nor, *a fortiori*, cyanogen. It therefore contains carbon, and from these two determinations its atomic weight, or equivalent, would have the half of the quantity of carbon contained in cyanogen. It is perhaps identical with argon, of which about 1 per cent has been found in atmospheric air, and which would therefore be a *carbide of nitrogen*, containing half the carbon contained in cyanogen.

Laboratory of Analytical Chemistry,
Putney, S.W.

LONDON WATER SUPPLY.

Report on the Composition and Quality of Daily Samples of the Water Supplied to London for the Month Ending April 30th, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To Charles Perrin, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, May 10th, 1900.

Sir,—We submit herewith, at the request of the Directors, the results of our analyses of the 184 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from April 2nd to April 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 184 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The drought recorded last month still continues, and was more accentuated during the latter half of the month, when only 0·08 inch of rain fell from the 13th to the 30th inclusive. The total rainfall at Oxford during April was 0·89 inch; the average is 1·66 inches, the deficiency is therefore 0·77 inch for the month. This reduces the excess for the year to 0·84 inch; at the present time the deficit is 11·8 per cent on the thirty years' average.

Our bacteriological examinations of 456 samples have given the results recorded in the following table; we have also examined 32 other samples, from special standpipes, &c., making 488 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 23 samples) ..	245
New River, filtered (mean of 101 samples) ..	4
Thames, unfiltered (mean of 23 samples) ..	955
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 251 samples)..	10
Ditto ditto .. .. highest	118
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 23 samples) ..	324
River Lea, from the East London Company's clear-water wells (mean of 35 samples) ..	5

The results of the bacteriological examinations may be summed up in stating that the London supply during the month has been of very high microbial quality, and the results of the filtration highly satisfactory.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

ELECTROLYTIC DETERMINATIONS AND SEPARATIONS.*

By LILY G. KOLLOCK.

(Continued from p. 222).

Mercury.

It is recorded (*Am. Chem. Journ.*, xi., 264) that mercury may be separated without difficulty with a low current and a large excess of alkaline cyanide. The deposits are noted as "compact, rather grey in colour, and showed in a few cases a drop-like nature so characteristic of mercury." The time required for the deposition under the conditions given was twelve to fourteen hours. Smith and Wallace (*Journ. Am. Chem. Soc.*, xvii., 612) published a number of separations of mercury from other metals—zinc, cadmium, cobalt, and nickel. The time required for separation was reduced to three and three and one-half hours.

In the experiments made this year the following conditions gave good results and confirmed those previously made:—A solution of mercuric chloride, containing 0·1439 grm. of mercury in 10 c.c. and five-tenths grm. of potassium cyanide, was diluted to 100 c.c. It was electrolysed with a current $N.D._{100}=0·07$ A and V=3·2. The temperature of the solution was 65° C. In three hours the metal was completely deposited. The deposit was washed with slightly warm water (see Table X.).

The deposits obtained were light grey in colour and were easily washed. They were bright and metallic in appearance with higher voltage.

Separations.

Mercury from Copper.—Smith and Frankel (*Am. Chem. Journ.*, xi., 264) report the separation of mercury and

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxi., No. 10.

TABLE X.

Mercury. Grm.	Potassium cyanide. Grm.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury found. Grm.
0·1439	0·5	100	N.D.$_{100}$=0·07 A.	3·2	65°	3½	0·1441
0·1439	0·5	100	,, =0·07 A.	3·2	65°	3½	0·1442
0·1439	0·5	100	,, =0·02 A.	1·6	65°	6	0·1440
0·1439	0·5	100	,, =0·07 A.	3·1	65°	3	0·1436

TABLE XI.

Mercury. Grm.	Copper. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury found. Grm.
0·1216	0·1	2·0	125	N.D.$_{100}$=0·02 A.	1·09	Cold	16	0·1220
0·1216	0·1	2·5	125	,, =0·03 A.	1·9	65°	4	0·1216
0·1216	0·15	3·5	125	,, =0·035 A.	1·8	65°	2½	0·1213
0·1216	0·1	3·0	125	,, =0·04 A.	1·9	65°	3	0·1215

TABLE XII.

Mercury. Grm.	Nickel. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury found. Grm.
0·1216	0·1	2	125	N.D.$_{100}$=0·04 A.	1·7	65°	4	0·1220
0·1216	0·1	2	125	,, =0·04 A.	2·2	65°	4	0·1220
0·1216	0·15	2·5	125	,, =0·04 A.	2·2	65°	4	0·1211
0·1216	1	2	125	,, =0·04 A.	2·2	65°	4	0·1213

TABLE XIII.

Mercury. Grm.	Cobalt. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury deposited. Grm.
0·1216	0·1	2	100	N.D.$_{100}$=0·025 A.	2·45	Cold	16	0·1217
0·1216	0·1	2	100	,, =0·025 A.	2·06	65°	5	0·1213
0·1158	0·1	2	100	,, =0·03 A.	2·9	65°	5	0·1152

TABLE XIV.

Mercury. Grm.	Zinc. Grm.	Potassium cyanide. Grm.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.
0·1158	0·1	2	125	N.D.$_{100}$=0·05 A.	3	60°	4	0·1152
0·1158	0·1	1·5	125	,, =0·033 A.	2·5	50°	4	0·1155
0·1158	0·2	2	125	,, =0·025 A.	2·9	50°	4	0·1155

TABLE XV.

Mercury. Grm.	Platinum. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury deposited. Grm.
0·1373	0·025	3	125	N.D.$_{100}$=0·05 A.	2·12	65°	4	0·1372
0·1373	0·01	3	125	,, =0·04 A.	2·12	75°	4	0·1376
0·1373	0·01	3	125	,, =0·045 A.	2·12	70°	4	0·1375

TABLE XVI.

Mercury. Grm.	Iron. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury deposited. Grm.
0·1216	0·1	1	100	N.D.$_{100}$=0·05 A.	2	70°	3	0·1215
0·1216	0·3	3	100	,, =0·04 A.	2	70°	4	0·1217
0·1216	2·5	2·5	100	,, =0·02 A.	2·7	65°	4	0·1217

copper in an alkaline cyanide solution. A current generating 2 c.c. of oxyhydrogen gas per minute was used for this purpose. The solution used contained 0·1833 grm. of mercury and 14—70 per cent of copper was added. With a current generating $3\frac{5}{10}$ c.c. of oxyhydrogen gas, mercury was completely deposited in sixteen hours. It is stated, however, that when the quantity of copper present exceeded 20 per cent the results were unsatisfactory. Later, Smith and McCauley took up the separation again, and by careful regulation of the current were enabled to effect it even when the metals were present in equal quantities.

In repeating this work, a solution of mercuric chloride equivalent to 0·1216 grm. of mercury and a copper sulphate solution equivalent to one-tenth grm. of copper were used. An excess of potassium cyanide was added. The entire solution was diluted to 125 c.c. The current acting upon the solution was N.D.$_{100}$=0·04 A and V=1·09. The temperature was 65°. In from two to four hours the mercury was completely deposited. On testing the metal qualitatively the deposit was found free from copper (see Table XI).

Mercury from Nickel.—These metals were separated in an analogous manner to that described under the separation of silver from nickel. To the solutions of mercuric chloride and nickel nitrate were added 2 grms. of potassium cyanide. This was then diluted to 125 c.c. A current of N.D.$_{100}$=0·04 A and V=2·2 was allowed to act for three hours. The deposit was found to weigh 0·0003 grm. less than the calculated amount of mercury present. The filtrate was found to contain no mercury. No nickel was deposited with the metal (see Table XII.).

Mercury from Cobalt.—The mercuric chloride used in this separation contained 0·1216 grm. of mercury, and the cobalt nitrate contained one-tenth grm. of cobalt. Two grms. of potassium cyanide were added to the mixed solutions, which were diluted to 100 c.c. The current acting

was N.D.$_{100}$=0·03 A and V=2·9. With a temperature of 60° the deposition of the mercury was complete in five hours. It was free from cobalt (see Table XIII.).

Mercury from Zinc.—The results obtained in the separation of mercury from zinc were first reported by Smith and Frankel in 1889. The electrolysis took place at the ordinary temperature in sixteen hours with a current generating 3 c.c. of oxyhydrogen gas per minute.

In the experiments made this year the following conditions gave satisfactory results (see Table XIV.).

In the third determination the current acted for three hours at 50°, and then all night in the cold. The deposit in all cases was grey and compact. The mercury on examination for zinc gave no reaction for that metal, while no mercury was found in the zinc solution.

Mercury from Cadmium.—As stated under the separation of silver from cadmium, Smith and Wallace (*Journ. Am. Chem. Soc.*, xvii., 612) found that cadmium might be separated from both silver and mercury by keeping the temperature slightly elevated. Unless this precaution is taken, cadmium is precipitated with both metals, even when a low current is used.

The following proved to be good working conditions:—The mercuric chloride solution used contained 0·1182 grm. of mercury, and the cadmium sulphate solution was equivalent to 0·2206 grm. of cadmium. The double alkaline cyanides were produced by the addition of 2½ grms. of potassium cyanide. A current of N.D.$_{100}$=0·018 A and V=1·7 was passed through the solution, previously heated to 65° for seven hours. The mercury deposit was free from cadmium and weighed 0·1181 grm., which was 0·0001 grm. lighter than the theoretical amount of mercury. It was repeatedly found that when the current was greater than 0·02 A, cadmium was precipitated.

Mercury from Platinum.—This separation was first carried out and reported by Smith (*Am. Chem. Journ.*, xiii., 417). The deposit of mercury was effected by using a current generating 2 c.c. of oxyhydrogen gas a minute. The alkaline cyanide used for 0·1902 grm. of mercury was 2½ grms. According to these suggestions a separation of the two metals was made. The mercury in solution was 0·1373 grm., and the platinum in the platinic chloride contained from 0·025 grm. to 0·1 grm. In four hours the separation was completed. The current was N.D.$_{100}$=0·05 A and V=2·1—2·3. The results show an error of −0·0001 grm. to +0·0003 grm. (see Table XV.).

Mercury from Iron.—The separation of mercury from iron, like that of silver from iron, has not been previously attempted in alkaline cyanide solution. Accordingly the following method was developed and found to yield satisfactory results:—

The solution of the cyanide of iron was prepared from ferrous ammonium sulphate in precisely the same way as that described in the separation of silver from iron. The mercuric chloride solution contained 0·1216 grm. of mercury. The cyanide present was from 1 to 3 grms., and the current was N.D.$_{100}$=0·05 A and V=2·5. The temperature during the electrolysis was maintained at 70° C. In three hours the mercury was completely deposited; it was free from iron. The mercury deposit was dissolved in concentrated nitric acid. After diluting, a few crystals of ammonium thiocyanate were added; a faint pink colour was observed, which was found to be due to the small trace of iron in the nitric acid (see Table XVI.).

(To be continued).

Detection of Glycerin.—L. Grunhut.—As the result of a number of comparative experiments, the author finds that there is but one certain method of detecting the presence of glycerin when it exists in only small quantities. A mixture of the substance under examination, and double its weight of SO_4KH, is made in a small flask: this is heated on a sand-bath; an escape-tube leads the fumes into a flask surrounded with a refrigerating mixture. If the substance contains glycerin the odour of acrolein is perceived very distinctly when cold.—*Zeit. Anal. Chem.*, xxxviii., p. 37.

ON THE PROGRESS OF CHEMISTRY IN GREAT BRITAIN AND IRELAND DURING THE NINETEENTH CENTURY.*

Part I.—From the Beginning of the Century to the Foundation of the Chemical Society (1841).

By T. E. THORPE, LL.D., F.R.S.,
President of the Chemical Society.

(Continued from p. 224).

The promulgation of the atomic theory and the importation of the ideas of constancy of chemical composition and of definite numerical ratio, gave an immediate impetus to analytical chemistry, of which the operations were at length seen to be subject to a numerical check and control hitherto unknown and barely suspected. Whilst the reflecting goniometer, on the one hand, served to define the geometrical form of a substance, and to establish the constancy of that form with a precision unattainable by the graphometer of Carangeau, the balance, on the other, rendered more and more perfect, in response to the demands of the chemist upon the mechanician, in order to satisfy the conditions imposed by the fundamental laws of Dalton's hypothesis, served to determine its exact atomic composition. The first use to which Wollaston, in 1810, put his reflecting goniometer, was to measure with exactitude the primary angles of calcspar, the composition of which had been accurately ascertained by Richard Phillips in 1803. A number of compounds, indeed, had their real nature and composition established during this period, and in the light of the atomic theory. In fact, the memoir by Thomson on the Oxalates (*Phil. Trans.*, 1808), and that by Wollaston "On Super-acid and Sub-acid Salts" (*Phil. Trans.*, 1808), were of the greatest service as proving the validity of Dalton's theory of chemical combination. Smithson Tennant analysed emery. Hatchett ascertained the composition of magnetic pyrites; Chenevix that of the arseniates of copper and iron, and of bournonite; Thomson that of sodalite, fluor-spar, and allanite.† Edward Clarke, of Cambridge, analysed petalite, gehlenite, the purple of Cassius, and examined the newly-discovered metal cadmium and its salts.

It would be impossible to exaggerate the influence which the discoveries of the first twenty years of the century exerted on the spirit of the age. They seemed, indeed, to presage a new era—the coming of a glorious day of which the dawn had already begun. No generalisation was more opportune in its announcement than that of Dalton; it was, indeed, to use the common phrase, "in the air." The teaching of a long succession of philosophers and the experimental labours of many workers had paved the way for its acceptance, and it is practically certain that had Dalton not formulated it, Wollaston or Berzelius would have done so. As it was, each of these distinguished men was almost immediately able to supply the strongest experimental proof of its soundness. Wollaston, from the outset, was well aware of the limitations of Dalton's explanation of the facts of chemical combination; and he recognised that the arithmetical relation alone of the proportions of elementary atoms is insufficient to explain their mutual action. As he says, "we shall be obliged to acquire a geometrical conception of their relative arrangement in all the three dimensions of solid extension." How Berzelius received the news of Dalton's discovery he has himself related in his memorable paper in *Gilbert's Journal*. This view of the combinations of bodies, he says, "appeared capable of illustrating so greatly the doctrine of affinity, that the confirmation of Dalton's hypothesis seemed to be the greatest step that chemistry, as a science, would have made during the whole time of its existence." How it was confirmed by Berzelius it is not

* Presidential Address to the Chemical Society, 1900.

† Allanite, in addition to cerium, contained, as he supposed, a new metal which he proposed to distinguish by the name of "junonium."

necessary here to state, nor need we set out in detail how the whole course of modern chemistry, however complex and many-sided it may seem, is really one vast elaboration of the atomic theory; in fact, as Liebig has said, all our ideas are so interwoven with that theory that it is difficult to carry ourselves back to the time when it did not exist. Dalton was the first recipient of one of the Royal medals which the Sovereign, on the recommendation of the Royal Society, annually bestows, and no more just award was ever made. Davy, in making known, as he said, "this first testimony of royal benevolence to science," stated that this discovery of the simple principle, universally applicable to the facts of chemistry, laid the foundations for future labours respecting the sublime and transcendental parts of the science of corpuscular motion. Dalton's merit, in this respect, resembles that of Kepler in astronomy. "The causes of chemical change are as yet unknown, and the laws by which they are governed; but in their connection with electrical and magnetic phenomena, there is a gleam of light pointing to a new dawn in science; and may we not hope that in another century, chemistry having, as it were, passed under the dominion of the mathematical sciences, may find some happy genius, similar in intellectual powers to the highest and immortal ornament of this Society, capable of unfolding its wonderful and mysterious laws."

Volta's famous letter of March 20th, 1800, to Sir Joseph Banks, was as the "order of the day" of a great leader. No more momentous document was ever given to the scientific world. "The voltaic battery," said Davy, in one of his characteristically happy phrases, "was as an alarm-bell to experimenters in every part of Europe; and it served no less for demonstrating new properties in electricity, and for establishing the laws of this science, than as an instrument of discovery in other branches of knowledge; exhibiting relations between subjects before apparently without connection, and serving as a bond of unity between chemical and physical philosophy."

It is not necessary here to dilate upon what this "instrument of discovery" in the hands of our countrymen has done for the development of chemistry. April 30th, 1800, when Nicholson and Carlisle first effected the decomposition of water by electrolysis, is a red-letter day in the history of our science. The *Transactions of the Royal Society*, *Nicholson's Journal*, and the *Philosophical Magazine* in the first years of the century show with what ardour and with what a lively expectancy "the new galvanic apparatus of Mr. Volta" was applied.*

Although even as far into the century as the date of Waterloo, the theory of the compound nature of water was regarded by some as "nothing more than a fanciful conjecture" and "a bare assertion without any proof whatever," there can be no question that Nicholson and Carlisle's discovery was of the greatest service in affording what was then, and long subsequently, looked upon as one of the strongest analytical proofs of the validity of the fact. But in their effect upon the popular imagination, these discoveries altogether paled before that of Davy. The isolation of potassium is, in reality, one of the most dramatic episodes of an epoch fruitful in striking incidents. The remarkable nature of the substance itself, its intense chemical activity, its significant chemical relationships, and the astonishing and far-reaching possibilities which its very existence seemed to open out, were all calculated to impress the minds of *virtuosi* and vulgar alike. Davy's triumph, indeed, was regarded as the realisation of a confident anticipation that the most important discoveries in chemistry were about to be made by the help of galvanism—an anticipation which found expression in almost every issue of the scientific journals of the time. There is little wonder, therefore, that a succession of scientific events of such magnitude as those which characterised the opening years of this century should have powerfully excited the imaginations of all cultured persons, and that optimists in every country, under the sway of ideas which made up the social and political philosophy of the time, should look forward in sure and certain hope in the immediate advent of a new era—to the coming of a generated humanity in a golden age. Thus Christopher Girtanner, of Göttingen, whose name lives only in connection with the fact that he was the first German chemist to appreciate and teach the new doctrines of the French School, was convinced that in the 19th century the transmutation of the metals would be generally known and practised. "Every chemist," he says, "every artist, will make gold; kitchen utensils will be of silver, and even gold. . . . There will be no other riches than natural riches—the productions of the soil; artificial riches, such as gold, silver, and paper money, will vanish in the hands of those who have accumulated them. What a revolution in society! Every enlightened chemist, however, will agree with me that this revolution is not only probable, but at no great distance." Unhappily for Dr. Girtanner's reputation as a prophet, the social revolution he foretold may not be reckoned among the achievements of the 19th century. Enlightened chemists still appear to have an eye to artificial riches, not scorning even paper money; and although, unfortunately, the precious metals do occasionally vanish in our hands, it is not because we convert them into kitchen utensils.

Even the sober-minded Faraday was not insensible to the visions thus created. In the course of his lectures on the metals, given in 1818 to the City Philosophical Society, he says, "to decompose the metals, then to re-form them, to change them from one to another, and to realise the once absurd notion of transmutation, are the problems now given to the chemist for solution. Let none start at the difficult task, and think the means far beyond them. Let us but look to the means which have given us these bodies, and to their gradual development, and we shall then gain confidence to hope for new and effective powers for their removal from the elementary ranks. . . . . Consider the improvement when, by a variety of manipulations, the early chemist of the last century separated a small quantity of a metallic substance from five or six other bodies, where it existed in strong combination, and then passed to the perfection of these means as exhibited in the admirable researches of Tennant and Wollaston; lastly, glance but at the new, the extraordinary powers which the chemist of our own nation put in action so successfully for the reduction of the alkalis and earths, and you will then no longer doubt that powers still progressive and advanced may exist and put at some favourable moment the bases of the metals in our hands."

It is interesting to note that, even as far back as 1806, an effort was made to form a Chemical Society independent of the Royal Society, to be called the London Chemical Society, but it received scant encouragement from persons in high places, and few chemists of note joined its ranks. Sir Joseph Banks, indeed, frowned down upon all such attempts. "I see plainly," he once said, "that all these new-fangled associations will finally dismantle the Royal Society, and not leave the old lady a rag to cover her." And the frown of the masterful old President meant social ostracism to all who chose to disregard it. But, unlike his predecessor, Sir John Pringle, who, on a certain memorable occasion, confessed to George III. that he was unable to reverse the laws and operations of nature, and was then told he had better resign, Banks, if he could not reverse, could at least control and modify a natural tendency, and with the aid of Wollaston, Davy, Hatchett, and Brande, he managed to keep chemistry for a time almost exclusively under the cloak of "the old lady."

Priestley died on February 9th, 1804. Although he

* This at the time was usually constructed of plates of silver and zinc, with intervening pieces of cloth. "Most of our philosophers," we are told, "have used half-crowns for the silver plates," and zinc could be bought at 8d. per lb. at the White Lion in Foster Lane, and cast in moulds of chalk.

lived long enough to have read of Dalton's explanation of the laws of chemical combination, as explained in Thomson's "System," and although he must have heard of the electrolytic decomposition of water by Nicholson and Carlisle in 1800, nothing apparently could shake his conviction of the essential and inherent truth of the conception of phlogiston. The last of his published writings was his memoir on "The Doctrine of Phlogiston established, and that of the Composition of Water refuted." Tolerant and receptive as he was in all other matters of opinion, and especially in matters of religious belief, he seemed utterly incapable of appreciating the real significance of the rapidly accumulating mass of facts, or of drawing any correct inferences from them.

Cavendish, "le plus riche de tous les savans et probablement aussi le plus savant de tous les riches," died in 1810. His work belongs to a preceding age, for on the downfall of phlogistonism he had ceased to occupy himself with chemical pursuits, and it must ever remain doubtful how far he gave in his adhesion to the new doctrine to which his own cardinal discovery so largely contributed.

In the year 1815 appeared Thomson's *Annals of Philosophy* (vi., 321), an anonymous essay "On the Relation between the Specific Gravities of Bodies in their Gaseous State and the Weights of their Atoms," which the author states he submits "to the public with the greatest diffidence; for, although he has taken the utmost pains to arrive at the truth, yet he has not that confidence in his abilities as an experimentalist as to induce him to dictate to others far superior to himself in chemical acquirements and fame." With this modest preamble the author proceeds to discuss certain consequences which seem to him to follow from the doctrine of volumes at first generalised by Gay-Lussac. The paper is remarkable, in spite of its errors and crudities, for its originality and lucidity. Its author was eventually discovered to be William Prout, then a young medical student, who lived to become eminent as a physiological chemist. His work thus tentatively put forward has become classical as containing, or rather suggesting, the hypothesis that the atomic weights of the elements are multiples of that of hydrogen. For it is noteworthy that the paper nowhere contains in explicit terms the statement of what is now known as Prout's law. Its main purpose was to set out more clearly than had hitherto been perceived the relation between the doctrine of gaseous volumes and of atoms, and it is only incidentally remarked that he "had often observed the near approach to round numbers of many of the weights of the atoms," and that "Dr. Thomson appears to have made the same remark." He further points out that it would appear from his tables "that all elementary numbers, hydrogen being considered as 1, are divisible by 4, except carbon, azote, and barytium, and these are divisible by 2, appearing, therefore, to indicate that they are modified by a higher number than that of unity or hydrogen. Is the other number 16 or oxygen? And are all substances compounded of these two elements?" Prout's "law" has been frequently so stated as to imply that all substances are "compounded" of hydrogen. But, as will be seen, his own words convey no such idea.

It is not improbable that the idea that all atomic weights were actually integers would have attracted little, if any, attention, had it not been adopted by Thomson and given currency by means of his "System of Chemistry," then, and for some years subsequently, one of the leading British text-books.

Smithson Tennant, Professor of Chemistry in the University of Cambridge, a man of whom it was said, in Johnson's well-known words, is "to be mentioned with reverence rather for the possession than the exertion of uncommon abilities," died in 1815—accidentally killed whilst riding in the neighbourhood of Boulogne. Born at Selby, in 1761, and a pupil of Black, he was early attracted to the study of chemistry, which he continued to prosecute at Cambridge as a Fellow Commoner of Christ's College. His private fortune exempted him from the necessity of following actively any profession. Although a man of wide reading, and of a quick and active mind, we are told that there was a singular air of carelessness and indifference in his habits and mode of life, his manners, appearance, and conversation being the most remote from those of a professed student. "His college rooms exhibited a strange, disorderly appearance of books, papers, and implements of chemistry, piled up in heaps, or thrown in confusion together." He was fond of foreign travel, and, in the course of one of his journeys, made the acquaintance of Scheele, for whom he had a high admiration. He was also on terms of friendship with many of the leading French chemists of his time. Chemistry in Germany at this period was a mixture of science and credulity, the philosopher's stone was spoken of with respect, and Tennant relates that he received from a man of science and character an introduction to a person who was reputed to be in possession of that treasure, with whom he conversed in Latin, and who exhibited to him the mysterious powder "enlarging upon its transcendent qualities with much pomp, and in flowing and sonorous periods."

Tennant is best known for his analysis of carbon dioxide, his work on the nature of the diamond, and on the injurious effects of magnesia in agriculture, his chemical examination of emery, and his discovery of iridium—so-named from the various colours of its solutions, and osmium—which owes its name to the smell of certain of its oxides. In the course of an inquiry by a Committee of the Royal Society, formed at the request of the Government to investigate the danger that might attend the general introduction of gas lighting in the metropolis, he made the observation that flame will not pass through small tubes, a discovery which in the hands of Davy led to the invention of the safety lamp.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 3rd, 1900.

Professor Thorpe, F.R.S., President, in the Chair.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—Messrs. Alexander William Bain, B.A., Heinrich Rowland Beringer, Frank Curzon Britten, B.A., John Richard Brooke, A.I.C., Arthur Brand Chater, Nicholas Cullinan, M.D., John Cussons, B.Sc., Arthur Charles Davis, Nöel Fielding Deerr, William Donald, Robert Eadie, Frederic Richard Ellis, Thomas FitzGibbon, Morris Broad Fowler, Reginald Gordon Halstead, James Herbert Haines, B.Sc., Henry Jennings, James Henry Kershaw, Adolf Liebmann, M.A., Ph.D., Thomas Lamb Lockhart, Thomas Macara, Frederick Thomas Munton, Robert Pettigrew, Geoffrey Surtees Phillpotts, B.A., James Wallace Sandford, Bertram D. Steele, B.Sc., James Walter Tilley, Augustus John Walker, B.Sc.

Certificates were read for the first time in favour of Messrs. George Edward Battle, Totlands, Dane Park, Ramsgate; John Henry Gough, 22, Francis Street, Chapeltown Road, Leeds; Percy John Hinks, 16, Moreton Road, South Croydon; Tajiro Ichioka, 8, Nottingham Place, London; David Herbert Patrick, Earlsdon, Coventry; Timothy A. Smiddy, Kilbarry House, Cork, Ireland.

Of the following papers, those marked * were read:—

*61. "*The Substituted Nitrogen Chlorides and Nitrogen Bromides Derived from Ortho- and Para-acet-toluide.*" By F. D. Chattaway and K. J. P. Orton.

Both *o*- and *p*-acet-toluide when treated with hypochlorous or hypobromous acid, produce nitrogen chlorides and bromides, which readily undergo transformation into the isomeric substituted toluides; these in their turn form nitrogen chlorides and bromides, which pass into the disubstituted toluides, in which the amino-hydrogen is again replaceable by chlorine or bromine. As with the substituted anilides, when the ortho- and para-positions relatively to the acetamino-group are occupied, the compounds undergo no further isomeric change, hydrolysis, or decomposition resulting from attempts to induce it.

The halogen, exactly as in the case of acetanilide or a substituted acetanilide, is found always to take up the para-position when this is possible, and when this position is occupied, the ortho-position, relatively to the carbon to which the nitrogen is attached. The authors have shown (*Trans.*, 1899, lxxv., 1046; 1900, lxxvii., 134; *Ber.*, 1899, xxxii., 3573), that substitution by chlorine or bromine in anilines and anilides is not a direct process, but a result of the transformation of a nitrogen chloride or bromide, a hypochlorite or hyprobromite being probably formed first by the nitrogen becoming quinquevalent. The behaviour of the nitrogen chlorides and bromides derived from the toluides is in entire agreement with this conclusion.

The properties of the nitrogen chlorides and bromides obtained from the toluides resemble exactly those of other members of the class. The nitrogen chlorides are colourless, the nitrogen bromides sulphur-yellow solids, all crystallising well in four-sided prisms terminated by domes.

All show the general reactions of nitrogen chlorides and bromides. They react with alcohol, re-forming the toluide and producing ethyl hypochlorite or hypobromite, although this action is usually accompanied by a considerable amount of isomeric change.

They react with ammonia liberating nitrogen, with hydrocyanic acid forming cyanogen chloride or bromide, with hydrogen chloride, bromide, or iodide setting free the halogen, and with hydrogen peroxide oxygen, in each case the toluide being re-formed. On warming with dilute acids, a certain amount of hydrolysis accompanies the isomeric change, and this, in the case of the nitrogen bromides, is the chief action.

The following compounds are described:—

o-*Methyl-phenyl-acetyl-nitrogen chloride* (ortho-acet-chloramino-toluene), $CH_3 \cdot C_6H_4 \cdot NCl \cdot COCH_3$, m. p. 43°.

Methyl-chlor-phenyl-acetyl-nitrogen chloride (ortho-acet-chloramino-5-chlor-toluene), $CH_3 \cdot Cl \cdot C_6H_3 \cdot NCl \cdot COCH_3$ (Me : Cl : : 2 : 4), m. p. 66°.

Methyl-dichlor-phenyl-acetyl-nitrogen chloride (ortho-acet-chloramino-3 5-dichlortoluene),—

$$CH_3 \cdot Cl_2 \cdot C_6H_2 \cdot NCl \cdot COCH_3$$

(Me : Cl : Cl : : 2 : 4 : 6), m. p. 78°.

p-*Methyl-phenyl-acetyl-nitrogen chloride* (para-acet-chloramino-toluene), $CH_3 \cdot C_6H_4 \cdot NCl \cdot COCH_3$, m. p. 91—92°.

Methyl-chlor-phenyl-acetyl nitrogen chloride (para-acet-chloramino-3-chlor-toluene), $CH_3 \cdot Cl \cdot C_6H_3 \cdot NCl \cdot COCH_3$ (Me : Cl : : 4 : 2), m. p. 48°.

Methyl-dichlor-phenyl-acetyl nitrogen chloride (para-acet-chloramino-3 : 5-dichlor-toluene),—

$$Cl_2 \cdot CH_3 \cdot C_6H_2 \cdot NCl \cdot COCH_3$$

(Me : Cl : Cl : : 4 : 2 : 6), m. p. 72°.

o-*Methyl-phenyl-acetyl-nitrogen bromide* (ortho-acet-bromamino-toluene), $CH_3 \cdot C_6H_4 \cdot NBr \cdot COCH_3$, m. p. 100·5°.

Methyl-brom-phenyl-acetyl-nitrogen bromide (ortho-acet-bromamino-5-brom-toluene), $CH_3 \cdot Br \cdot C_6H_3 \cdot NBr \cdot COCH_3$ (Me : Br : : 2 : 4), m. p. 91°.

Methyl dibrom-phenyl-acetyl-nitrogen bromide (ortho-acet-bromamino-3 : 5-dibromotoluene),—

$$CH_3 \cdot Br_2 \cdot C_6H_2 \cdot NBr \cdot COCH_3$$

(Me : Br : Br : : 2 : 4 : 6), m. p. 120°.

p-*Methyl-phenyl-acetyl-nitrogen bromide* (para-acet-bromamino-toluene), $CH_3 \cdot C_6H_4 \cdot NBr \cdot COCH_3$, m. p. 64—95°.

Methyl-brom-phenyl-acetyl-nitrogen bromide (para-acet-bromamino-3-brom-toluene), $Br \cdot CH_3 \cdot C_6H_3 \cdot NBr \cdot COCH_3$ (Me : Br : : 4 : 2), m. p. 87°.

Methyl-dibrom-phenyl-acetyl-nitrogen bromide (para-acet-bromamino-3 : 5-dibromo-toluene),—

$$Br_2 \cdot CH_3C_6H_2 \cdot NBr \cdot COCH_3$$

(Me : Br : Br : : 4 : 2 : 6), m. p. 118°.

°62. "*The Estimation of Hypoiodites and Iodates; and the Reaction of Iodine Monochlorides with Alkalis.*" By K. J. P. Orton and W. L. Blackman.

The method of estimating hypoiodites is based on the oxidation of sodium arsenite by hypoiodites, but not by iodates. Excess of the standard arsenite is used, and the unoxidised portion titrated with iodine after the saturation of the alkali by carbon dioxide. The iodate can then be determined by acidifying and titrating the iodine liberated. The hypoiodite solutions were prepared chiefly from iodine monochloride (dissolved in hydrochloric acid), and also from iodine solution. The initial reaction of iodine monochloride and alkalis is represented by the equation $ICl + 2MOH = MIO + MCl + H_2O$.

With potash, soda, lime-water, and baryta water, the whole of the iodine is for a short time (varying from thirty seconds to five minutes) present as hypoiodite. The transformation into iodate and iodide is practically complete after twenty-four hours. With ammonia, the complete transformation requires two or three weeks. The solution obtained from methylamine and iodine monochloride behaves in an exceptional manner, the hypoiodite gradually disappearing, probably oxidising the methylamine. The bleaching of indigo is shown to be unsuitable for the purpose of estimating hypoiodites, owing to the direct action of even dilute alkalis on the indigo.

The solutions obtained by shaking up iodine with mercuric oxide and water contain chiefly iodate, and only a small quantity of hypoiodite.

Discussion.

In reply to the President, Dr. Orton said that the work of which he had given an account was complete in fifteen months before the abstract of R. L. Taylor's paper (*Proc.*, 1900, xvi., 70) appeared.

°63. "*Products of the Action of Sulphur Dioxide on Ammonia.*" By Edward Divers.

When the paper on "Ammonium Amidosulphite" (Divers and Ogawa, *Proc.*, 1900, xvi., 38; *Trans.*, 1900, lxxvii., 327) was communicated to the Society, the authors were unaware that a paper on the subject of this note had been published by Schumann (*Zeit. Anorg. Chim.*, 1900, xxiii., 43). Beyond the fact that Schumann also points out the incorrectness of Rose's statement that sulphur dioxide combines with only its own volume of ammonia, the two papers have nothing in common. The main fact established in the paper by Ogawa and the author was the existence of ammonium amidosulphite, of which they had already given notice in a paper on "Ethyl Ammoniumsulphite" (*Trans.*, 1899, lxxv., 534). Schumann has obtained a product of the same composition as ammonium amidosulphite in red masses, not very hygroscopic; it dissolves in water to a yellow solution, forming ammonium sulphate, sulphite, thiosulphate, trithionate, and pentathionate, whilst ammonium sulphite is a white, crystalline powder extremely deliquescent, and soluble in water, forming ammonium sulphite only. Schumann obtained two other products, having respectively the formulæ SO_2,NH_3 and $3SO_2,4NH_3$, the aqueous solutions of which were similar to that given by the red substance $SO_2,2NH_3$. The author can only regard Schumann's three products as mixtures of substances formed by the spontaneous decomposition of ammonium amidosulphite.

Since the publication of the paper on ammonium amidosulphite, further work by Ogawa has fully established

the existence amongst the products of the decomposition of this salt of the compound formulated (*loc. cit.*, p. 334) as $(NH_3)_2S_2O_3$, but of which the true formula is $N_2H_4S_2O_3$. Although only faintly acid to litmus, it forms salts, of which the *ammonium salt*, $NH_4N_2H_3S_2O_3$, the *potassium salt*, $KN_2H_3S_2O_3$, the *barium salt*, $Ba(N_2H_3S_2O_3)_2,2H_2O$, and the *lead salt*, $Pb(N_2H_3S_2O_3)_2$, have been prepared. The constitutional formula $NH_2{\cdot}S{\cdot}NH{\cdot}SO_3H$, or—

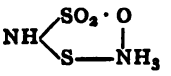
$$NH\begin{matrix}<SO_2\cdot O\\ S\text{——}NH_3\end{matrix},$$

seems to express the relations of this substance as far as they are at present known.

This preliminary notice is published because, unfortunately, the circumstances in which the author and Ogawa are placed afford little hope of the full account of the products of the decomposition of ammonium amidosulphite being ready for publication for some considerable time to come.

Discussion.

Dr. Travers asked if the author attributed the formula—



to the compound he had described merely from analogy with amidoacetic acid.

Dr. Hewett asked whether any decomposition products of the compound $N_2H_4S_2O_3$ had been obtained in which the sulphur and nitrogen were still linked together. There seemed to be a possibility of isolating the sulphur analogue of hydroxylamine, or some compound nearly related to it.

Dr. Divers replied that this substance hydrolysed as a sulphonic acid, and yet was not acid to litmus like all other sulphonic acids. The red substance soluble in carbon disulphide and ether might well be the sulphur amide, $S(NH_2)_2$, supposed to exist by Forchhammer.

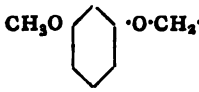
64. "*On Brazilin.*" IV. By A. W. Gilbody, W. H. Perkin, jun., and J. Yates.

In a previous communication (*Proc.*, 1899, xv., 27), it was shown that trimethylbrazilin, $OH{\cdot}C_{16}H_{10}O(OCH_3)_3$, when oxidised with chromic acid, is converted into *trimethylbrazilone*, $OH{\cdot}C_{16}H_8O_2(OCH_3)_3$, and that this substance when heated loses water, with formation of *dehydrotrimethylbrazilone*, $OH{\cdot}C_{16}H_6O(OCH_3)_3$, a compound which yields an acetyl derivative, and which is evidently identical with the acetyl compound which Herzig prepared by the oxidation of acetyltrimethylbrazilin (*Monatshefte*, 1895, xvi., 913). The authors now find that trimethylbrazilone, on treatment with phenylhydrazine, is reduced to a substance having the formula $C_{19}H_{18}O_4$, which melts at 173°, and dissolves in nitric acid and other concentrated mineral acids, yielding intense purple or orange solutions.

When trimethylbrazilin is oxidised with potassium permanganate under various conditions, a variety of substances are formed, the principal of which are the following:—

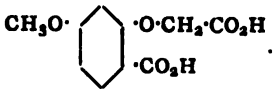
(1). A dibasic acid, $C_{10}H_{10}O_6$, melting at 175°. This acid contains one methoxyl group, and is derived from the resorcinol portion of the molecule of brazilin, because, when fused with potash, it yields a substance which gives an intense violet colouration with ferric chloride.

When heated with water at 200°, this acid, $C_{10}H_{10}O_6$, is decomposed, with elimination of carbonic anhydride and formation of a monobasic acid, $C_9H_{10}O_4$ (m. p. 119°), which has been shown by synthesis to have the constitution—

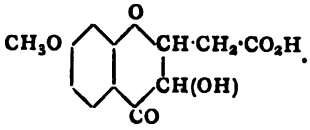


In synthesising this acid, for which the name *methylresorcinolacetic acid* is proposed, pure methylresorcinol, $CH_3O{\cdot}C_6H_4{\cdot}OH$, was treated with sodium ethoxide and ethyl bromacetate, and in this way *ethyl-methylresorcinolacetate*, $CH_3O{\cdot}C_6H_4{\cdot}O{\cdot}CH_2{\cdot}CO_2Et$, was obtained as a thick, colourless oil boiling at 182° (25 m.m.); this, on hydrolysis, yielded the corresponding acid which melted at 119°, and was found to be identical with the acid $C_9H_{10}O_4$, obtained from brazilin as described above.

Since brazilin under other conditions (see below, and *Proc.*, 1899, xv., 27—29) yields *p*-methoxy-salicyclic acid it follows that the acid, $C_{10}H_{10}O_6$, must be the *methylcarboxyresorcylacetic acid* having the formula—



(2). A monobasic acid, $C_{10}H_8O_3(OCH_3){\cdot}CO_2H$, melting at 129·5°, which has already been described (*Proc.*, 1899, xv., 29). We find that this acid, when treated with concentrated sulphuric acid at 80°, loses water, and is converted into an acid, $C_{12}H_{10}O_5$, which melts at 196°, and when oxidised with permanganate yields *p*-methoxysalicylic acid. It is therefore probable that the acid, $C_{10}H_8O_3(OCH_3){\cdot}CO_2H$, has the constitution—



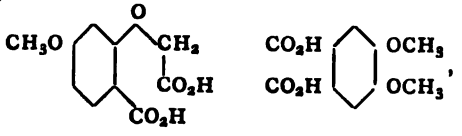
(3). An acid very sparingly soluble in water and melting at 208°. This acid has the formula $C_{19}H_{18}O_9$; it yields a *silver* salt $C_{17}H_{16}O_5(CO_2Ag)_2$, and a *dimethyl* salt, $C_{17}H_{16}O_5(CO_2Me)_2$ (m. p. 110°), and is therefore dibasic. When fused with potash, it yields a substance soluble in water, which, with ferric chloride, gives an intense catechol reaction; it does not form an oxime, but when reduced with sodium amalgam, it is converted into an acid, $C_{17}H_{16}O_4(CO_2H)_2$, which melts at 227°.

When the acid $C_{17}H_{16}O_5(CO_2H)_2$ is heated at 140° with alcoholic potash, it is decomposed with formation of a neutral substance, the analysis of which agrees better with the formula $C_{17}H_{20}O_5$ than with $C_{17}H_{18}O_5$; it appears probable therefore that, besides elimination of carbonic anhydride, reduction has taken place according to the equation $C_{17}H_{16}O_5(CO_2H)_2+2H=C_{17}H_{20}O_5+2CO_2$. This substance crystallises from alcohol in needles, melts at 140°, and appears to be a lactone.

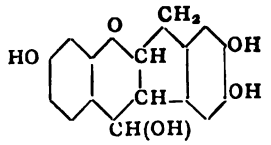
(4). An acid very sparingly soluble in water, which melts at 214—215° with vigorous decomposition, and gives also an intense catechol reaction after fusion with potash and treatment with ferric chloride. The results of one analysis point to this acid having the formula $C_{13}H_{14}O_8$, but, unfortunately, it has so far been obtained in such small quantity that the analysis could not be repeated.

(5). Metahemipinic acid.

Constitution of Brazilin.—The results described in this and previous communications, and especially the isolation of methylcarboxyresorcylacetic acid and of metahemipinic acid,—



from among the products of the oxidation of the brazilin, allow of only a very few formulæ as possible expressions of the constitution of this substance, and after carefully considering all the facts, the authors are of the opinion that brazilin probably has the constitution represented by the formula,—



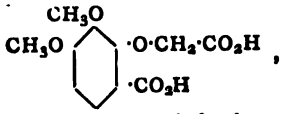
65. "*On Hæmatoxylin.*" V. By W. H. Perkin, jun., and J. Yates.

When *tetramethylhæmatoxylin*, $OH\cdot C_{16}H_9O(OCH_3)_4$, is oxidised by potassium permanganate under various conditions, it yields the following substances:—

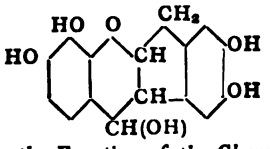
(1). *Metahemipinic acid*, which was characterised by analysis and by conversion into the characteristic ethylimide melting at 230°. The formation of this acid from tetramethyl-hæmatoxylin by oxidation with chromic acid has already been mentioned (*Proc.*, 1899, xv., 241).

(2). An acid melting at 180°, which evidently corresponds with the acid, $C_{17}H_{16}O_5(CO_2H)_2$ (m. p. 208°) obtained from trimethylbrazilin as described in the preceding abstract. This acid has the formula $C_{18}H_{18}O_6(CO_2H)_2$, and is dibasic, since it yields a silver salt $C_{18}H_{18}O_6(CO_2Ag)_2$. It dissolves in concentrated sulphuric acid with a crimson colour, and when reduced with sodium amalgam is converted into an acid, $C_{18}H_{18}O_5(CO_2H)_2$, which melts at 194°.

(3). An acid melting at 214°, which when fused with potash, is converted into a substance which with ferric chloride gives the pyrogallol reaction. This acid has the formula $C_{11}H_{12}O_7$, and is dibasic, since its silver salt has the composition $C_{11}H_{10}Ag_2O_7$. It contains two methoxyl groups, and when heated with concentrated hydrochloric acid at 200° it yields pyrogallol. When treated with acetic anhydride, it gives an anhydride, $C_{11}H_{10}O_6$ (m. p. 154°); it therefore has the constitution:—



and corresponds with the methylcarboxyresorcylic acid obtained from brazilin. The formation of this acid and of metahemipinic acid from hæmatoxylin leads us to believe that the constitution of this substance is represented by the formula—



66. "*Note on the Function of the Characteristic Meta-orientating Groups.*" By Arthur Lapworth.

The author desires to draw attention to the following points, the significance of which does not appear to be generally recognised.

The groups $\cdot CO\cdot R$, $\cdot NO_2$, $\cdot CN$, $\cdot SO_2R$ are known to produce the following effects when in direct attachment to—

(1). *A benzenoid nucleus.*—They render the hydrogen atom in the *meta* position directly replaceable by all the characteristic substituting agents, thus differing from all other known groups.

(2). *A* :CH *group.*—They render the hydrogen atom directly replaceable, and the compounds exist in the tautomeric forms $:CH\cdot X:Y$ and $:C:X\cdot YH$.

(3). *A* $\cdot CH_2$ *group.*—The compounds are usually capable of condensation with aldehydes, or of entering into the Claisen reaction with esters of oxalic, formic, and nitrous acids in presence of sodium ethoxide, that is to say, under conditions favourable to, if not actually productive of, the "iso"-forms of the compounds in question.

The connection between the foregoing facts is clearly brought out by the use of the mode of analysis suggested by the author (*Trans.*, 1898, lxxiii. 446).

The Claisen reaction is not applicable to nitrobenzene, in which there is no $\cdot CH_2$ group, but should, in accordance with the author's view of the function of the above groups when attached to a benzenoid nucleus (*loc. cit.*), be applicable to benzenoid compounds containing a $\cdot CH_2$ group in the *ortho-* or *para*-position to one of the groups in question.

Reissert (*Ber.*, 1897, xxx., 1030) states that whilst the methyl group of *o-* and *p*-nitrotoluene reacts with ethyl oxalate in presence of alcohol and sodium, no condensation occurs if ethyl nitrite is used. The author finds, however, that reaction occurs if alcohol-free sodium ethoxide and amyl nitrite are employed, and under these conditions *o*-nitrotoluene readily affords *o*-nitrobenzaldoxamine, which on hydrolysis and treatment with potash and acetone yields indigo. *p*-Nitrotoluene also reacts, though less readily, with amyl nitrite, affording *p*-nitrobenzaldoxime. If amyl formate be used in these reactions, condensation appears to take place, but the products are more difficult to isolate.

The fact that *m*-nitrotoluene does not react in this respect like the *ortho-* and *para*-compounds is in strict accordance with the author's views. It is hoped that it will be possible to show that the other *meta* orientating groups behave in the same manner as the nitro-group.

This occasion may be taken to point out that the application of recent views regarding the nature of unsaturated compounds (Thiele, *Annalen*, 1899, cccvi., 87) to the case of substitution in the benzenoid series appears to lead inevitably to the conclusion, already arrived at by the author (*loc. cit.*), that substitution in the *meta*-position, relatively to one of the above groups, is probably preceded by the formation of a tautomeric compound analogous to the "iso"-forms of the nitro-, cyano-, and keto-paraffins.

PHYSICAL SOCIETY.

Ordinary Meeting, May 11th, 1900.

Prof. O. J. Lodge, F.R.S., President, in the Chair.

A discussion of Prof. Lodge's paper "*On the Controversy concerning Volta's Contact Force*," was commenced by Prof. Armstrong.

Prof. Armstrong expressed his indebtedness to the President for putting forth clearly what we are trying to understand, and said that it was hardly time for chemists to enter the discussion when physicists themselves differed. There has apparently been a change in front, since the time when the effect was supposed to be due either to (1) chemical action between metals, or (2) oxidation. Prof. Lodge's view is intermediate, but approximates to the second. Prof. Armstrong said that from a practical point the existence of the effect was unknown because sufficient precautions had never been taken to prevent chemical action. He urged the continuance of experiments similar to those carried out by Mr. Spiers, and stated that modern ideas of chemistry were favourable to the view which Prof. Lodge had taken up with regard to the volta effect.

Mr. Glazebrook made some remarks upon the meaning of the term E which occurs in the expression for the Peltier effect at the junction of two metals. If we confine our attention to an infinitesimal cycle at the junction of two metals at slightly different temperatures we get the equation for the Peltier effect in which E is the potential difference at the point considered. If then, assuming reversibility, we sum up all the infinitesimal cycles round a circuit, and get a finite cycle, the E M.F. of the circuit is a function of the two temperatures between which it is working. Differentiating with respect to temperature the total E.M.F. of the circuit, we get an equation which applies to the circuit as a whole, and in which E is the total E.M.F. round the circuit.

Mr. Price asked if any critical experiment could be suggested to settle the question.

Dr. Lehfeldt called attention to some experiments which had been performed to measure the potential difference between an electrolyte and a gas. The electrolytes considered were chiefly aqueous solutions, and the potential differences observed varied largely. The surface tensions of the liquids were measured, and it was shown that the variations in the potential difference were very similar to those in surface tension. This suggests, in the case of electrolytes, true physical surface effects, and not chemical action.

The Chairman remarked that Dr. Lehfeldt evidently looked upon the metal-ether boundary as being the effective one. The experimental evidence is not sufficient to say exactly which is the effective contact, but it seems to show that the metal-ether effect is of the same order of magnitude as the oxygen layer effect. According to Helmholtz they ought to be related, and they apparently are.

The Chairman then read a paper by Mr. J. B. Tayler on "*The Heat of Formation of Alloys.*"

Experiments have been made upon alloys of lead with tin, bismuth, and zinc, and of zinc with tin and mercury. The method employed consisted in dissolving (1) the alloy, and (2) the corresponding mixture of metals in mercury and measuring the heat of solution in each case. On the assumption that the solutions obtained are identical, the difference between the heat of solution of the mixture and that of the alloy is the heat of formation of the latter. The calorimeter was a thin glass tube silvered on the outside, and supported by a stouter tube silvered on the inside. Suitable arrangements were adopted for the introduction of the metals or alloys, which were used in the form of filings. Solution was often complete in less than a minute, and rarely took more than two minutes and a half. The alloys first experimented upon contained their constituents in equivalent proportions, and the heats of formation were found to be small in comparison with those found for brass by Galt and Baker. It was thought that only a small percentage of the atoms present had entered into definite chemical combination, and that more reliable results would be obtained by dissolving a small quantity of one metal in an excess of the other, and calculating from the experimental results the heat of formation of the grm.-molecular weight of compound upon the supposition that the whole of the small quantity of metal had entered into chemical combination by the exercise of its normal valency. Using the numbers so obtained to find, by Kelvin's theory, the potential difference which should exist between the metals concerned when put in contact, results were arrived at which agreed neither with the Volta effect or the Peltier effect, but which were considerably nearer the former than the latter.

A paper on "*The Want of Uniformity in the Action of Copper-zinc Alloys on Nitric Acid*" was read by Dr. J. H. Gladstone.

Experiments have been made by dissolving copper-zinc alloys in nitric acid, following the method of Dr. Galt, and adopting the precautions mentioned by him. The reaction between nitric acid and these metals or alloys is very complicated, and there is a difference between the products in the case of an alloy and in the case of the equivalent mixed metals. The gases evolved being small in the experiments performed, attention was directed to the determination of the substances remaining in solution, *i.e.*, the nitrous acid and ammonia. The alloys gave much more nitrous acid and less ammonia; in fact, two of the alloys employed produced no ammonia. Discrepancies in results may be due to the fact that the zinc and copper in contact form a zinc-copper couple, which in the presence of acid sets up a vigorous action, and produces a different evolution of heat. Difficulties arise in the investigation because the alloys used may not be definite chemical compounds, but mixtures of two or more alloys with uncombined zinc and copper. The alloy with 38·38 per cent of copper appears to be fairly uniform. Different observers disagree as to the amount of heat produced by any reaction, but the excess of calories in a zinc reaction over those in a copper reaction appears to be fairly constant. Starting with 640 calories, the value, according to Galt, when copper is dissolved in nitric acid of sp. gr. 1·360, we should have 1357 calories when zinc is dissolved, provided the chemical action is the same in each case. All the calorimetrical results from the different specimens of alloys would theoretically lie upon the straight line drawn between 604 and 1357. This is practically so from pure copper to the copper 70 per cent alloy, but beyond that there is less heat produced than that indicated by the straight line law, the maximum deviation lying at about copper 37 per cent. The specimen containing 38·38 per cent copper, which is not far from the alloy $CuZn_2$, shows a loss of 32 calories. The only way in which this deficit can be accounted for is by supposing that the action of this alloy on nitric acid produces a larger quantity of nitric oxide then in the case of pure copper. But allowing full force to this argument it cannot account for as much as 10 calories of the deficit. There is therefore a residual deficit as yet unaccounted for on chemical grounds. The author states that it is desirable that experiments should be conducted on the zinc-copper alloys with solvents which give a simpler chemical action than that produced by nitric acid.

The Chairman pointed out that the results obtained by Galt for an alloy which appeared to be a chemical compound were in close agreement with what would be expected from the existence of the Volta contact force.

Prof. Armstrong said that the action of nitric acid on brass or zinc and copper was a function of the quantity of acid present, its strength, the temperature, and the pressure, and that therefore it was unsatisfactory to conduct experiments using nitric acid as a solvent. He suggested the use of a solution of bromine, in which finely powdered zinc, copper, and brass are easily soluble with a simple chemical reaction.

Mr. Tomlinson pointed out that it was impossible to use the ordinary formula for the calculation of the Volta effect from the heat of formation of alloys, unless we know exactly the chemical composition of the alloy which is produced.

Mr. W. R. Cooper, referring to Mr. Tayler's paper, said that it was difficult to see that anything could be proved by the application of the Kelvin theory to a metallic contact unless there is ground for believing that some particular alloy of fixed composition is always formed. There is also a further difficulty in converting heat of formation into E.M.F. in cases where the metals have different valencies, for there is no reason why one valency should be selected rather than the other. Referring to Dr. Gladstone's paper, Mr. Cooper said that it was possible that the difference in the reducing powers of mixtures and alloys might be due to local action, which would be more pronounced in the case of alloys. More hydrogen would be evolved, and the reduction would be more complete.

Prof. S. P. Thompson then showed an Electromagnetic Experiment. A circular coil, capable of carrying a strong current, was placed with its axis horizontal in a tank of water. Into the tank were also placed some small magnets in sealed glass tubes, so adjusted as to have a density approximately equal to that of water. The magnets just floated or just sank. On running a current through the coil it was possible to "fish" for the magnets, which, acted upon by the magnetic field, immediately made their way to the coil. When the current was carefully reversed upon the approach of a magnet, repulsion instead of attraction took place, and the magnet retreated. In general, however, reversal of the current produced reversed polarity in the magnet, and attraction still persisted.

The Society then adjourned until May 25th.

CORRESPONDENCE.

DETERMINATION OF NICKEL IN NICKEL STEEL.

To the Editor of the Chemical News.

Sir,—Although Mr. Sargent in his paper on "The Determination of Nickel in Nickel Steel" (Chem. News, lxxxi., p. 209) standardises the potassium cyanide with the *nitrate* of nickel, he remarks later that "the titration may be accomplished in the presence of most all the salts of ammonium and the fixed alkalis *except the nitrates*."

The titration Mr. Sargent employs is very extensively used under the impression that alkaline nitrates exert no appreciable influence; if that impression does not correspond with actual fact, for ammonium nitrate at least, then many others besides myself have frequently returned inaccurate results.

To remove the uncertainty which Mr. Sargent's assertion originates I have titrated 0·05 grm. of nickel in the presence of varying amounts of the undermentioned alkali nitrates. The figures (c.c. KCN) used show that nitrates do not interfere at all.

Cryst. nitrate of—	0	5	10	20 grms. used.
Ammonium ..	33·8	33·75	33·8	33·7 c.c.
Sodium	33·8	33·8	33·8	33·75 ,,
Potassium ..	33·8	33·8	33·8	33·7 ,,

—I am, &c., J. T.

OBITUARY.

Dr. EDMUND ATKINSON.

It is with regret that we record the death, on the 4th inst., of Dr. Edmund Atkinson, who, as one of the older generation of scientific men, was well known as a chemist and physicist. He was born at Lancaster in 1831, and in his early days acted as assistant to the late Sir Edward Frankland in the laboratory of Owens College, Manchester. He was also a member of the staff of Queenwood College when that school was distinguished by the presence of a band of teachers which included Professors Tyndall, Hirst, Frankland, and Dr. Debus, the latter of whom alone survives. Afterwards Dr. Atkinson was for many years Professor of Experimental Science at the Royal Military College, Sandhurst, and at the Staff College, Camberly. After he retired he continued to live at Camberly, and, as a magistrate and in other ways, he took an active part in the management of local affairs. Dr. Atkinson was perhaps best known as the translator of Ganot's "Physics." He was buried at Woking Cemetery on the 8th inst.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 16, April 17, 1900.

Heat of Combustion of Very Volatile Liquids.—MM. Berthelot and Delépine.—The combustion of organic substances can be effected with ease and precision in the calorimetric bomb by means of oxygen compressed to 25 atmospheres. Very volatile bodies, however, need special precautions to avoid partial vaporisation in the bomb, when oxidation commences, and false calorimetric numbers result. The authors overcame these difficulties. A small glass bulb is weighed and then filled as completely as possible with the volatile liquid. After sealing this is again weighed and placed in the bomb by the side of a small weighed piece of camphor and a little gun-cotton (about 0·02 gr.) in contact with a platinum wire. After closing the bomb and admitting the compressed oxygen the gun-cotton is fired by the wire and causes the little glass bulb to explode and the combustion of the products at the same moment. By this means the heats of combustion of the ethyl aldehyde, methylal, methyl formate, ethyl formate, propylic aldehyde, and isopropylic aldehyde were determined.

New Microchemical Reaction of Palladium.—M. E. Pozzi-Escot and H. C. Conquet.—If a solution of palladium chloride and potassium nitrite are mixed and immediately afterwards excess of a caustic alkali is added (potash, soda, or ammonia), beautiful rhombohedral crystals are formed of a yellow colour, due to the formation of a double nitrite of palladium and potassium. All elevation of temperature must be avoided, since this causes a decomposition of the molecule. To obtain good crystals evaporation must be very slow.

MEETINGS FOR THE WEEK.

Monday, 21st.—Society of Arts, 8. (Cantor Lectures). "The Incandescent Gas Mantle and its Use," by Prof. Vivian B. Lewes.

Tuesday, 22nd.—Royal Institution, 3. "Brain-Tissue considered as the Apparatus of Thought," by Alex. Hill, M.A., M.D.

—— Society of Arts, 8. "The Practice of Lettering," by Edward F. Strange.

Wednesday, 23rd.—Society of Arts, 8. "Salmon Legislation," by J. Willis-Bund.

Thursday, 24th.—Royal Institution, 3. "Chaucer," by the Rev. Canon Ainger, M.A., LL.D.

—— Society of Arts, 4.30. "English Criminal Procedure and the Indian Code of Criminal Procedure—a Comparison," by Sir John Scott, K.C.M.G., D.C.L.

Friday, 25th.—Royal Institution, 9. "The Great Alpine Tunnels," by Francis Fox, J.P., M.Inst.C.E.

—— Physical, 5. Experiments Illustrating the Aberration called Coma, by Prof. S. P. Thompson, F.R.S. "Notes on the Measurement of some Standard Resistances," by R. T. Glazebrook, F.R.S. "On the Strength of Ductile Materials under Combined Stresses," by J. J. Guest.

Saturday, 26th.—Royal Institution, 3. "The Growth of Chamber Music" (with Musical Illustrations), by Sir Frederick Bridge, Mus. Doc.

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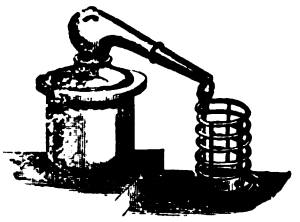

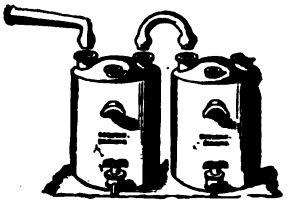

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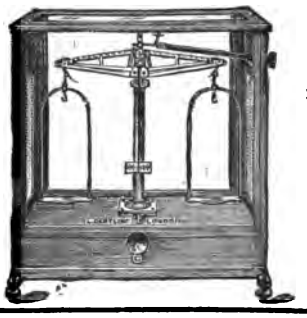

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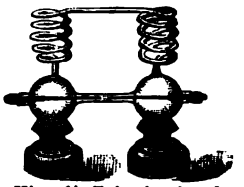

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VOL. LXXXI., No. 2113.

ON CERTAIN PROPERTIES OF THE ALLOYS OF GOLD AND COPPER.*

By Professor Sir W. C. ROBERTS-AUSTEN, K.C.B., F.R.S., and T. KIRKE ROSE, D.Sc.

THE alloys of gold and copper, which are of great industrial importance owing to their use in coinage, have not been subjected hitherto to systematic examination. It has been assumed that they differ widely from the silver-copper series, which has been studied from different points of view, but there is very little evidence on which this view can be based.

Examination with the aid of a thermo-couple and autographic recorder shows that the freezing-point curve of the gold-copper series consists of two branches setting out from the points of solidification of the pure metals and meeting at a point, which is the freezing-point of the eutectic. The eutectic contains about 82 per cent of gold, and 18 per cent of copper, or about 60 atoms of gold to 40 of copper, and solidifies at 905°. The general shape of the curve therefore resembles that of the silver-copper series when the abscissæ give the relative number of atoms.

Under the microscope, alloys containing more than 82 per cent of gold show a minutely granular structure in which it is not certain that two constituents can be distinguished. The section of standard gold containing 91·6 per cent of gold bears a close resemblance to that of standard silver prepared in the same way. The alloy with 80 per cent of gold shows the characteristically-banded eutectic structure almost exclusively, and the alloys with less gold consist of crystals of copper set in a matrix of the eutectic.

Another point of similarity between the gold-copper and silver-copper series is that both the eutectics are brittle, and show scarcely any extensibility; they differ in these respects from most other eutectics. Analysis of various portions of ingots of standard gold reveals the fact that liquation takes place as definitely as in standard silver, the difference in composition between the centre and the outside of similar ingots being, however, three or four times greater in standard silver than in standard gold. In the latter case, the centre contains from 0·3 to 1·0 part per 1000 less gold than the outside.

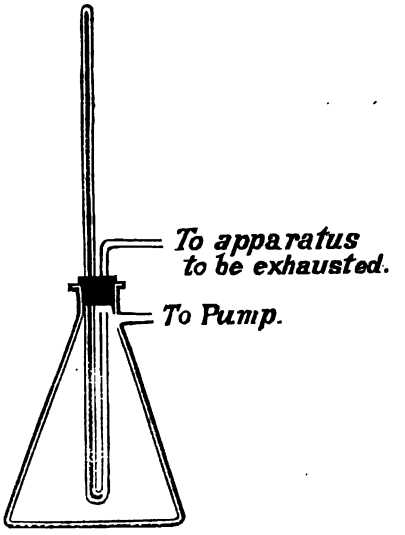
It follows from these results that the gold-copper series of alloys presents many points of similarity with the silver-copper series, and that the main difference is only one of degree, copper being apparently more soluble in gold in the solid state than in silver.

A SIMPLE AND PORTABLE GAUGE FOR VACUUM PUMP.

By E. G. BRYANT.

I HAVE found this contrivance, which is almost sufficiently described by the sketch, of considerable use, especially in lecture experiments. Its chief advantages are its portability and the difficulty of upsetting it.

The gauge is simply a U-tube with the open end long enough to reach nearly to the bottom of the safety-flask or bottle attached to the pump itself. The longer end passes through one of the holes in the cork, and is closed at the top. The length of this limb depends, of course, on the depth of the flask. With a 16-oz. flask the two limbs can be about 7 and 15 to 17 inches respectively. Enough mercury is introduced into the tube to fill the longer end and cover the bend, or the whole tube may be filled, and the excess will run out in use and collect in the flask itself. The open end is kept close up against the cork to prevent water being splashed into it. By arranging that the bend shall be about an inch from the bottom of the flask, the gauge may be pushed down whenever the



mercury becomes hidden by the cork. The tube should be fairly strong, to resist the sudden jerk of the mercury when the pressure is relieved.

In order to obtain a gauge of the full barometric height, it would be necessary to have the short limb about 2½ times the diameter of the long one. Messrs. P. Harris and Co., Lim., of Birmingham, have arranged to supply such a tube; the longer limb about 1 m.m. internal diameter, and both limbs graduated similarly to a syphon barometer.

Pontefract, May 12, 1900.

THE ESTIMATION OF BENZOL IN COAL-GAS.

By O. PFEIFFER.

THE volumetric analysis of gases does not allow of the separation of the gaseous hydrocarbides from the fumes of the heavy hydrocarbides which, grouped under the name of ethylene and benzol, are the most important with regard to illuminating gas. Among the numerous methods which have been proposed, that of Harbeck and Lunge is the most to be trusted (*Zeit. Anorg. Chemie*, 1898, xvi., 41). It is based on the separation of the benzol as a bi-nitrated product; the following is the method of procedure:—With the aid of an aspirator fitted with a thermometer and a manometer about 10 litres of the gas are passed through the nitrating mixture. The acid gases are absorbed by tubes filled with caustic potash or soda. The nitrating mixture is poured over a layer of ice, neutralised with soda, and treated with ether. After driving off the solvent, the dinitrobenzol is dried either by heating or *in vacuo*, and weighed. But as the nitrating mixture absorbs a part of the carbonic oxide, this method does not give altogether exact results, and it becomes necessary to modify it in the following manner:—The

* Abstract of a Paper read before the Royal Society, May 10, 1900.

apparatus, for both the measurement and the nitration of the gas, consists of a separating funnel of 3 or 4 litres capacity, accurately calibrated. After having introduced the gas, the temperature and pressure are noted, and 10 c.c. of the nitrating mixture are run in; this mixture consists of equal parts of nitric acid, of 1·52 density, and concentrated sulphuric acid. When the absorption of the benzol fumes is complete, 100 c.c. of water are run into the funnel, well shaken, and transferred to a smaller separating funnel, and the acid neutralised by means of 40 grms. of crystallised soda. It is then treated two or three times over with 50 c.c. of ether, and, after having got rid of the solvent, the dinitrobenzol is dissolved in absolute ether; the liquor is then filtered through a layer of calcined soda and collected in a small crystallising jar. After having evaporated the ether the residue is dried over sulphuric acid. To transform the weight of the dinitrobenzol into per cent (by volume) of benzol vapour we use the formula—

$$\frac{36080}{J} \cdot g \frac{273 + t}{b} = \text{per cent benzol vapour};$$

in which J represents the capacity of the large separating funnel, g the weight found, t the temperature, and b the pressure. We may also estimate the benzol vapour, with very close results, in the following manner:—We must first know the specific gravity, s, of the heavy carbides of hydrogen—a constant which can be obtained from the specific gravity, S, of the gaseous mixture, and from the proportion (per cent) in CO, CH_4, . . . &c., according to the formula—

$$s = 100S - (CO \cdot 0{\cdot}9621 + CH_4 \cdot 0{\cdot}5530 + H \cdot 0{\cdot}0692 + N \cdot 0{\cdot}9701 + CO_2 \cdot 1{\cdot}5197 \frac{1}{C_nH_m}.$$

By calling the total volume of the heavy hydrocarbides v, the benzol vapour contained in the gas = $\frac{(5 - 0{\cdot}9674)\, v}{1{\cdot}7367}$ per cent by volume.—*Journal für Gasbeleuchtung and Wasserversorgung*, xlii., 697.

ON THE ALKALOIDS OF AUSTRALIAN PLANTS.*

By G. HARKER, B.Sc.

Before beginning this investigation it was necessary to become conversant with the known methods for the extraction of alkaloids from plants and their subsequent purification. In obtaining information respecting the alkaloids in Australian plants already investigated, I was specially indebted to Mr. Maiden, whose papers I also made use of.

The following plants were examined:—

Monimieæ.—*Daphnandra micrantha* (Bentham). This plant has been examined by Dr. Bancroft physiologically and found to be active. In this investigation a considerable quantity of an alkaloid was isolated by me, and a large number of tests applied. The alkaloid, which so far as could be seen was amorphous, was present in the bark, the leaves containing a glucoside.

Laurineæ.—*Cryptocarya obovata* (R. Brown). No alkaloid present.

Pittosporeæ.—*Pittosporum undulatum* (Andrews). No alkaloid.

Euphorbiaceæ.—*Phyllanthus ferdinandi* J. Mueller). This plant contained a small trace of a volatile alkaloid.

In the case of each plant the bark and leaves were examined carefully. A most careful search was made for either fixed or volatile alkaloid, and large quantities of material were worked upon so that small traces should not be missed.

* Abstract of a Paper read at the Melbourne Meeting of the Australasian Association for the Advancement of Science, Jan., 1900.

ESTIMATION OF ARSENIC IN METALS AND ALLOYS.

By A. HOLLARD and L. BERTIAUX.

This method is based on the separation of arsenic by distillation; though not entirely new it has the advantage over similar methods of being applicable in the presence of a considerable quantity of antimony, without requiring more than one distillation, and further, it is very rapid. The apparatus used is the same as that already described in the paper on the commercial analysis of copper (*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 8).

5 grms. of the granulated metal are placed in the flask with 50 grms. of ferric sulphate freed from all traces of nitrous fumes by evaporation with an excess of sulphuric acid. Then by means of the funnel 150 c.c. of pure hydrochloric acid are run in, and the tap turned off, after seeing that the oil-bath containing the U-tube is at 150—175°; the flask is then gently heated. The metal dissolves, and the arsenic distils over in the form of arsenious chloride; this chloride is collected in the test-jar, which already contains 50 c.c. of water. The operation is stopped when 35 c.c. of liquid have passed into the test-jar; this will take about half an hour from commencing to heat the flask. Under these conditions the arsenic only passes into the test-jar, the antimony remains in the U-tube which contains glass beads; this U-tube also prevents the passage of any projections which might leave the flask.

The arsenious solution is then titrated with iodine. This method, however, is not absolutely correct when applied to the estimation of arsenic in metallic antimony, as 0·0015 grm. of antimony passes over on distillation.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 8.

THE DETECTION AND ESTIMATION OF FORMIC ALDEHYDE.

By C. NEUBERG.

The author has observed that *p*-dihydrazinodiphenyl, $NH_2.NH.C_6H_4.C_6H_4.NH.NH_2$, unites with formic aldehyde, giving a methylenic compound—

$$CH_2{=}N.NH.C_6H_4.C_6H_4.NH.N{=}CH_2$$

in the form of a yellow precipitate consisting of microscopic needles. This precipitate forms rapidly in the cold, and instantaneously at 60°. It is insoluble in organic liquids of the most varying characters, in alkalis, and in alkaline carbonates. It is rapidly destroyed by mineral acids, and acetic acid. It melts towards 220°, and decomposes at 240°. To prepare this body the author recommends the gradual addition of the solution of formic aldehyde at 40 per cent to an aqueous solution, heated to 60°, of hydrochlorate of *p*-dihydrodiphenyl; this latter salt must be absolutely pure and colourless, otherwise there will be strongly coloured accessory products formed.

The precipitate appears even when there is only 1/50000th part of formic aldehyde in solution, but it is necessary in such a case to wait for a few minutes.

Furfurol, which has some of the same reactions as formic aldehyde, does not give this one any more than do the other aldehydes and ketones. If one of these latter happens to be present, two volumes of alcohol should be added, so as to dissolve the compound it might form with the dihydrazine. The precipitate must then be treated with alcohol until the foreign matter is entirely removed.

Commercial hydrochlorate of dihydrazinodiphenyl may be used for qualitative work, but for estimation the cooled pure salt must be used, and the liquid containing the formic aldehyde added with continuous stirring; it is then gradually heated to 60° and left to stand, then filtered, washed with boiling water, alcohol, and dry ether, and

dried at 90°. The bright yellow colour should remain. For the titration to be exact the solution ought to contain 1 to 2 per cent of formic aldehyde. The results are always approximate, but the method is of advantage when there are other aldehydes and ketones present. We might also titrate volumetrically the excess of dihydrazine after filtering the precipitate.—*Berichte*, xxxii., p. 1961.

LABORATORY METHOD FOR THE CONTINUOUS AND UNIFORM GENERATION OF ACETYLENE, AND FOR ITS PURIFICATION.*

By J. A. MATHEWS.

For most purposes in which it is desired to employ a stream of acetylene, no purification is necessary, provided the gas has been properly generated from the usual quality of commercial carbide now being produced. Analyses from various sources show that the impurities in acetylene from this source rarely amount to 1 per cent, and a good part of this is likely to be nitrogen. The combined amounts of hydrogen sulphide, hydrogen phosphide, and ammonia seldom exceed 0·2 per cent. In order that the amount of impurities may be as small as possible it is necessary that the gas be produced in the cold. This cannot be done when water is allowed to drip slowly upon the carbide, and furthermore this method causes the gas to be given off spasmodically, and the lime residue often bubbles or froths to such an extent that solid matter in considerable amounts may be carried over with the gas. In obviating all these difficulties the following method has proved successful:—

The fragments of calcium carbide are placed in a wide-mouthed bottle. They may, with advantage, be suspended in a basket of coarse wire netting, which will keep the carbide away from the lime residue, and also allow it to be withdrawn at any time. The carbide is covered with an abundance of absolute or 95 per cent alcohol, and the bottle closed with a two-hole stopper. Through one hole water can be led from a reservoir bottle, and in the other an outflow tube is fitted. If 95 per cent alcohol is used, the water therein serves to start the generation of acetylene and to expel the air from the apparatus. If, now, water be added drop by drop, it comes slowly into contact with the carbide, and a steady production of acetylene results. Unless the amount of alcohol present is very small and the rate of generation very rapid, no material rise in temperature ensues. After the addition of water has been stopped, the evolution of gas may be allowed to proceed until the bubbling ceases; the alcohol may then be decanted from the residue and distilled, and, rejecting the first few cubic centimetres, the rest of the distillate will be nearly absolute alcohol.

If it is desired to purify the acetylene, any one or more of several methods may be employed. No single purifying agent which has as yet been advocated meets all the requirements. For general convenience and thoroughness the following combination of methods is suggested:—

The acetylene generated as described above is passed into a solution of 15·6 parts of crystallised copper sulphate in 100 parts of water, to which is added 5 parts of dilute sulphuric acid (Hempel and Kahl, *Zeit. Angew. Chemie*, liii., 1898), 1 volume of sulphuric acid to 4 volumes of water. This solution, besides retaining any vapour of alcohol and also the ammonia and hydrogen sulphide, is especially useful for absorbing hydrogen phosphide. As an additional safeguard the acetylene is next passed through one or two towers filled with coarse bits of pumice stone which have been saturated with an acetic or sulphuric acid solution of chromic acid (Ullmann, *J. für Gasbeleucht.*, 1899, xlii., 198, 374), or, in place of a second tower, a strong sulphuric acid solution of chromic acid may be used. By this means any of the three chief impurities, which might escape absorption in the copper sulphate solution, will be completely oxidised. A small amount of these purifying materials will serve for a large volume of gas, and the acetylene should issue both dry and pure and possessing a faint and agreeable odour. A return of the strong irritating odour indicates that the purifiers have become exhausted.

The purification of acetylene by means of moist chloride of lime or sodium hypochlorite, as suggested by Odernheimer (*Chem. Zeit.*, xxii., 21) and Lunge (*Zeit. Angew. Chem.*, 1897, p. 651) or as modified by Wolff* (*Chem. Zeit.*, xxii., 281), is impracticable because of the instability of the solutions and because chlorine and chlorination products of acetylene are produced, which must then be removed by lime. Frank's method (*Zeit. Angew. Chem.*, 1898, p. 1050), using strongly acid solutions of copper or iron chloride, does not seem very efficient, nor is the employment of bromine as a purifier convenient for laboratory use. The combination of Hempel's and Ullmann's methods seems to meet all the requirements and is easily and conveniently arranged.

* Contributions from the Havemeyer Laboratories, Columbia University, No. 16. *Journal of the American Chemical Society*, vol. xxii., No. 2.

THE CARBIDE OF GOLD.†

By J. A. MATHEWS and L. L. WATTERS.

Explosive compounds resulting from the action of acetylene or coal-gas upon alkaline solutions of cuprous' argentous, and mercuric salts have been known for many years. These compounds are for the most part flocculent precipitates which retain water with more or less tenacity, and at temperatures which will render them anhydrous they are apt to explode violently. The earlier investigators of these acetylides did not recognise them as true carbides, and their analyses show the presence of hydrogen and oxygen, which are reported as constituent atoms of the molecule. Berthelot (*Ann. Chim. Phys.* [4], ix., 425), who did a great deal of work upon acetylene derivatives, speaks of copper acetylide as "cuproacetyloxide," and gives it the formula $(C_2Cu_2H)_2O$. Reboul (*Comptes Rend.*, liv., 1229) gives silver acetylide as $(C_2HAg)_2+Ag_2O$, and Blochmann (*Ann. Chem.*, Liebig, clxxiii., 176, 177) gives respectively $C_2H_2Cu_2O$ and $C_2H_2Ag_2O$. All of these might better be written C_2Cu_2 or C_2Ag_2 plus more or less water. Miasnikoff (*Ann. Chem.*, Liebig, cxviii., 330) gives silver acetylide as $C_2H_2Ag_2$. Keiser (*Am. Chem. J.*, xiv., 285) showed, however, that when properly dried these two acetylides are simply C_2Ag_2 and C_2Cu_2, and later (*Ibid.*, xv., 535) produced mercuric acetylide, HgC_2, which is also hydrated at the time of its formation.

Berthelot (*Ann. Chim. Phys.* [4], ix., 425) seems to be authority for the statement which appears in a number of old chemical works, but for which these do not give him credit, that by passing acetylene through aurous thiosulphate, a yellow explosive precipitate is formed. He gives no analysis of the product, and does not give any suggestions as to its probable constitution.

We have recently investigated this interesting compound, which proves to be analogous to the copper and silver compounds, its formula being aurous carbide, Au_2C_2.

* Wolff treats the gas first to remove ammonia and then with hypochlorite, thus eliminating the danger of forming nitrogen chloride, which he says is possible.

† Contributions from the Havemeyer Laboratories, Columbia University, No. 17. Reprinted from the *Journal of the American Chemical Society*, vol. xxii., No. 2.

In the preparation of aurous sodium thiosulphate we followed the method of Fordos and Gélis (*Ibid.* [3], xiii., 394, 1845). Pure auric chloride was dissolved in 50 parts of water; 3·2 parts of sodium thiosulphate were dissolved in the same amount of water. The solution of gold was added to the thiosulphate very slowly and with constant stirring. A slight odour of sulphur dioxide was noticed, and a little sulphur separated.

The solution was filtered, and the sodium aurous thiosulphate was precipitated with absolute alcohol. It was not re-crystallised. The principal reaction is said to be $8Na_2S_2O_3+2AuCl_3=2Na_3Au(S_2O_3)_2+2Na_2S_4O_6+6NaCl$.

This compound forms a colourless precipitate, very soluble in water but insoluble in alcohol. To form aurous carbide an aqueous solution of the sodium aurous thiosulphate is made strongly alkaline with ammonia and a slow stream of acetylene passed through. The solution remains colourless for a short time and then becomes yellow, and finally a yellow flocculent precipitate appears. In this respect it differs from silver and copper carbides, which form almost instantly, and hence are used to test for the presence of minute quantities of acetylene. The precipitate is filtered off, washed with water and alcohol, and dried in a desiccator over sulphuric acid.

Properties.—When thoroughly dried, the carbide of gold is highly explosive either upon rapid heating, by a blow, or even by brushing with a camel's hair brush. The explosion generates sufficient heat to produce flame, and the gold is left in an extremely finely divided condition and black. Carbide of gold is easily decomposed by hydrochloric acid, giving acetylene and leaving a black residue of aurous chloride. The presence of acetylene was shown by passing it into ammoniacal silver nitrate, in which it produced silver acetylide. The aurous chloride was tested by boiling, which gave auric chloride and metallic gold. By boiling gold carbide with water it is decomposed into its constituents, no acetylene being produced. Cupric sulphate and neutral ferric chloride do not decompose it in the cold; when anhydrous it becomes darker in colour and of a brown tint, and if heated very gradually decomposes without explosion. This fact was made use of in the determination of gold. The sample to be analysed was heated in the air-bath very slowly until a temperature of 180° to 200° was indicated, and then ignited in the flame of a Bunsen burner. The black mass becomes yellow by the burning off of the carbon and annealing of the gold. The analyses resulted as follows:—

	Weight of sample.	Gold found.	Gold per cent.	Theory for Au_2C_2.
I.	0·029	0·0273	94·14	94·25
II.	0·0174	0·0164	94·25	94·25

One sample of aurous carbide after two days in the desiccator lost no weight at 100° C., nor was loss experienced on renewed heating up to 120° C. Another sample that had not stood so long over sulphuric acid lost 0·0004 grm. at 100°. By rapid heating in the air-bath explosions were obtained at various temperatures from 83° to 157° C. No attempt was made to secure especially uniform heat at all parts of the air-bath, but the thermometer bulb was in all cases very close to the dish containing the explosive compound. It seems then that the rate of heating has most to do with effecting the decomposition of gold acetylide. The violence of the explosion was shown by several unexpected explosions. In one case about 15 or 20 m.grms. of the substance were on a watch-glass. In trying to remove a small fragment with the point of a knife, the whole mass exploded with a sharp report, and the watch-glass was broken into a score of pieces. In another instance, while brushing some of the carbide from a filter-paper with a camel's hair brush into a dish, the particles adhering to the paper exploded with a loud report and flame, and the filter-paper was badly torn, but the bulk of the material, which was not over an inch or two away, was not exploded by the concussion. The same fact was noted in the first explosion above, in which case the watch-glass was standing beside a crucible containing quite a large amount of dry gold carbide.

No other aurous solutions were tested with a view to obtaining gold carbide. From auric chloride in aqueous solution metallic gold is precipitated by acetylene.

A solution of auric chloride made alkaline with potassium hydroxide does not give a precipitate under similar conditions, nor does an aqueous or ammoniacal solution of potassium auric cyanide give a precipitate when acetylene is passed into it.

ELECTROLYTIC DETERMINATIONS AND SEPARATIONS.*

By LILY G. KOLLOCK.

(Continued from p. 233).

Gold Separations.

Smith and Muhr (*Am. Chem. Journ.*, xiii., 417) report the separation of gold from palladium, platinum, zinc, cobalt, nickel, and copper.

Gold from Palladium.—It has been previously shown that palladium is not deposited with a current of 0·01 A in the presence of an excess of potassium cyanide. With a current liberating eight-tenths c.c. of oxyhydrogen gas a minute, and the solution containing $1\frac{1}{10}$ grms. of potassium cyanide, 0·1162 grm. of gold was deposited in from twelve to fourteen hours.

In accordance with this suggestion, the following conditions were obtained:—Two grms. of potassium cyanide were added to a solution of gold chloride containing 0·1256 grm. of metal and palladium chloride equivalent to one-tenth grm. of metallic palladium. This was diluted to 250 c.c. The current used was $N.D._{175}=0·06-0·09$ A and V = 1·5. The temperature during the decomposition was 65°. In five hours the current was raised to 0·09 A. The gold was found to be completely deposited and free from palladium (see Table XVII.).

Gold from Copper.—In the report referred to, the separation of gold from copper in cyanide solution took place with a current liberating eight-tenths c.c. of oxyhydrogen gas at the ordinary temperature in about twelve hours.

It was found that by heating the solution to 65° the precipitation was more rapid, but required more cyanide in order to keep the copper from being deposited with the gold. To a solution of gold chloride containing 0·1665 grm. of metallic gold and copper sulphate containing one-tenth grm. of copper were added 4 grms. of potassium cyanide. The solution (250 c.c.), warmed to 65°, was electrolysed with a current of $N.D._{175}=0·05-0·07$ A and V = 1·7 for two and one-half hours. In this time 0·1667 grm. of gold was obtained. There was no reaction for copper when the deposit treated with nitric acid was tested for that metal (see Table XVIII.).

Gold from Nickel.—The conditions under which this separation is made are similar to those obtained *under* the separations of silver and mercury from nickel. The solution of gold chloride used was equivalent to 0·1610 grm. of metallic gold, and nickel nitrate contained one-tenth grm. of nickel. They were converted into their double cyanides by the addition of 4 grms. of potassium cyanide. A current of $N.D._{100}=0·05$ A and V = 1·5 was passed through the solution (125 c.c.) at 60° for six hours. The gold on examination contained no nickel (see Table XIX.).

Gold from Cobalt.—More difficulty was experienced with the separation of these metals than with the pre-

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxi., No. 10.

TABLE XVII.

Gold. Grm.	Palladium. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Gold found. Grm.
0·1256	0·1	2	125	$N.D._{100}$=0·06—0·03 A.	2·5	65°	6	0·1250
0·1256	0·1	2	250	$N.D._{175}$=0·06—0·09 A.	2·5	65°	6	0·1257

TABLE XVIII.

Gold. Grm.	Copper. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Gold found. Grm.
0·1665	1	2	250	$N.D._{175}$=0·07 A.	1·7	65°	2½	0·1667
0·1627	1	2	250	,, =0·05—0·08 A.	1·9	65°	3	0·1627

TABLE XIX.

Gold. Grm.	Nickel. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Gold found. Grm.
0·1567	0·1	4	125	$N.D._{100}$=0·05 A.	1·7	65°	7	0·1567
0·1610	0·1	4	125	,, =0·05 A.	1·5	60°	7	0·1603

TABLE XX.

Gold. Grm.	Cobalt. Grm.	Cyanide. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Dilution. C.c.	Time. Hours.	Gold found. Grm.
0·1610	0·1	4	125	$N.D._{100}$=0·05—0·08 A.	1·7	60°	125	6½	0·1612
0·1610	0·1	4	125	,, =0·05—0·08 A.	1·7	60°	125	6	0·1608
0·1495	0·1	4	250	$N.D._{175}$=0·05—0·08 A.	1·7—2	60°	250	6	0·1497

TABLE XXI.

Gold. Grm.	Zinc. Grm.	Cyanide. Grms.	Current.	Voltage.	Dilution. C.c.	Temperature.	Time. Hours.	Gold found. Grm.
0·1495	0·1	4	$N.D._{100}$=0·06 A.	2·66	125	60°	7	0·1490
0·1608	1	4	$N.D._{175}$=0·06 A.	2·7	250	65°	7	0·1602

TABLE XXII.

Mercury. Grm.	Sodium sulphide (sp. gr. 1·19). C.c.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Mercury found. Grm.	Error. Grm.
0·1403	25	100	$N.D._{100}$=0·11 A.	2·5	70°	5	0·1400	0·0003
0·1403	25	100	,, =0·12 A.	2·7	70°	5	0·1404	0·0001

TABLE XXIII.

Copper. Grm.	Nitric acid. C.c.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Copper found. Grm.
0·1398	10	100	$N.D._{100}$=0·03 A.	1·7	Cold	16	0·1396
0·1398	10	100	,, =0·09 A.	1·9	70°	16	0·1397
0·1398	10	100	,, =0·09	1·9	65°	16	0·1398

ceding. Four grms. of potassium cyanide were added to the solution of gold chloride and cobalt nitrate equivalent to one-tenth grm. of cobalt. The current used for the deposition of the gold was $N.D._{100}$=0·05 A and V=1·7. The electrolysis was carried out at 60° C. It was found advantageous before the end to add 1 c.c. of a 20 per cent sodium hydroxide solution, and raise the current to 0·08 A, in order to deposit the last trace of gold. In this way the gold was entirely deposited in seven hours free from cobalt (see Table XX.).

Gold from Zinc.—These metals were successfully separated according to the following conditions:—Four grms. of potassium cyanide were added to a solution of gold chloride and zinc sulphate containing one-tenth grm. of zinc. The current acting upon the double cyanide was $N.D._{100}$=0·06 A and V=2·66. The temperature of the solution was 60°. In seven hours the deposition was complete. No zinc was found with the gold (see Table XXI.).

Gold from Platinum.—To the solution of gold chloride containing 0·1576 grm. of gold and platinic chloride equivalent to 0·125 grm. of platinum, were added $1\frac{5}{10}$ grms. of potassium cyanide. The volume of the solution was 250 c.c. A current of $N.D._{175}$=0·01 A and V=2·7 was used. The temperature was 70° C. In three hours the gold was entirely precipitated. No platinum was deposited with the gold. The deposit weighed 0·1479 grm.

Mercury from an Alkaline Sulphide Solution.—In 1891 Smith made the statement that mercury obtained as mercuric sulphide during analysis may be accurately estimated electrolytically. The sulphide was dissolved in sodium sulphide of sp. gr. 1·19, and electrolysed with a current generating 1 c.c. of oxyhydrogen gas per minute acting during the night. The mercury was always compact and grey in colour.

In working out conditions for the determination of mercury by this method, 25 c.c. of sodium sulphide were added

to a solution of mercuric chloride containing 0·1403 grm. of mercury. This solution, diluted to 125 c.c., was electrolysed at 70° C. by a current of $N.D._{100}=0·11$ A and V=2·5. In five hours the mercury was entirely deposited; it was light grey in colour. Slightly warm water was used in washing the deposit (see Table XXII.).

Copper.

"Moore advises dissolving the recently precipitated copper sulphide obtained during the ordinary course of analysis in potassium cyanide, and after the addition of an excess of ammonium carbonate, electrolyse the warm (70°) solution" (Smith's "Electro-chemical Analysis," p. 61). According to this suggestion, the following conditions for the determination of copper were found to give good results:—The copper sulphate solution used contained 0·1270 grm. of metallic copper; this was precipitated as sulphide, which was dissolved in 2 grms. of potassium cyanide and ammonium carbonate added. The solution, diluted to 125 c.c., was heated to 70° C. and electrolysed with a current of $N.D._{100}=0·02$—0·07 A and V=4. In three hours the copper was completely deposited. The copper was found to weigh 0·1269 grm.; that is, 0·0001 grm. less than the theoretical amount.

Copper from Nitric Acid Solution.—Copper has also been successfully determined electrolytically in a nitric acid solution (*Am. Chem. Journ.*, xii., 329). A solution containing 10 c.c. of concentrated nitric acid was diluted to 100 c.c. and electrolysed. The current used was $N.D._{100}=0·03$ A and V=1·7. At the ordinary temperature in sixteen hours 0·1396 grm. of copper was precipitated. The solution, treated with ammonium hydroxide, showed that the copper had been entirely deposited. With a temperature of 65° and a current of $N.D._{100}=0·09$ A and V=1·9 in five hours 0·1397 grm. of copper was found, and another determination under identical conditions gave the theoretical amount of copper present (see Table XXIII.).

Cadmium from Sulphuric Acid Solution.—Smith (*Am. Chem. Journ.*, ii., 42) describes the quantitative deposition of cadmium from its sulphuric acid solution. Cadmium oxide was dissolved in sulphuric acid. The solution contained 2 c.c. of free sulphuric acid of sp. gr. 1·095.

The volume of the solution was 25 c.c. In two hours the cadmium was completely precipitated. The amount of cadmium present was 0·0931 grm. The following conditions were found to give good results by this method:—A solution of cadmium sulphate containing 0·1599 grm. of metal, after adding 3 c.c. of sulphuric acid (sp. gr. 1·09) and diluting to 125 c.c., was electrolysed with a current of $N.D._{100}=0·078$ A and V=2·61. The temperature during the electrolysis was 65°. In five hours the deposition of cadmium was complete. The metal weighed 0·1604 grm.

(To be continued).

A "Thrift Chat."—A large number of the workpeople employed by the United Alkali Company, Ltd., attended a "Thrift Chat," which took place at St. Rollox Chemical Works, Glasgow, a few days ago. Mr. James M'Culloch presided, and a representative of the Glasgow Thrift Society gave an address, at the conclusion of which there was a distribution of thrift literature. The Thrift Chat owes its existence to the National Thrift Society, which some years ago hit upon the plan of holding friendly talks with the workpeople employed in factories, mills, foundries, shipbuilding yards, and so on, not in outside places of meeting, but in the works themselves. The talk usually takes place at the end of the mid-day meal hour. The plan is excellent, and the "Chats" are growing in popularity, not only amongst the workers but amongst the employers, who are giving the Society every facility for the dissemination of its helpful doctrines.

ON THE PROGRESS OF CHEMISTRY IN GREAT BRITAIN AND IRELAND DURING THE NINETEENTH CENTURY.*

Part I.—From the Beginning of the Century to the Foundation of the Chemical Society (1841).

By T. E. THORPE, LL.D., F.R.S.,
President of the Chemical Society.

(Continued from p. 235).

The third decade of the century saw the rise of Faraday, Graham, and Edward Turner. Faraday had thrown up book-binding in 1813, and attached himself to the Royal Institution, getting his "baptism of fire" in working upon Dulong's newly-discovered nitrogen chloride, which was then occupying Davy's attention. He assisted Davy, too, in his work on iodine, on the torpedo, on the nature of the diamond, on euchlorine, and on the chlorides of phosphorus, and in his memoir on "Some Combinations of Phosphorus" the master pays a well-merited tribute to the "accuracy and steadiness of manipulation of his assistant."

Although Davy had propounded his proofs of the elementary nature of chlorine as far back as 1810, and although these had been strengthened by his work on iodine in 1814, his doctrine was not universally accepted, the Scotch teachers in particular, notably Murray and Ure, even as late as 1818, making repeated and determined attempts to subvert it. Faraday did yeoman service in repelling the attack, which eventually degenerated into a wordy warfare between Murray and John Davy.

With the beginning of 1820 Faraday may be said to have served his apprenticeship to science. He had already contributed some two score notices and papers to the *Quarterly Journal of Science*, and had published his remarkable work with Stodart, a surgical instrument maker, on wootz and other alloys of steel. He had discovered tetrachlorethylene, perchlorethane, and ethylene diodide, an account of which constitutes his first paper in the *Philosophical Transactions*, and shortly afterwards he described, with Richard Phillips, a new compound of chlorine and carbon, first observed by Julin, of Åbo, and now known as hexachlorobenzene. In 1823 appeared his memorable paper "On Fluid Chlorine," and immediately afterwards were liquefied hydrogen chloride, sulphur dioxide, carbon dioxide, sulphuretted hydrogen, euchlorine, and nitrous oxide. It should be stated, however, that Monge and Clouet had already condensed sulphur dioxide, probably before the year 1800, and that Northmore, with the assistance of Accum, had obtained liquid chlorine in 1806 by compression at ordinary temperatures (*Nicholson's Journal*, 12 and 13). Northmore's description of the properties of liquid chlorine is so explicit that it is remarkable that the significance of his observation should have escaped notice. The name and fame of the Royal Institution are indissolubly connected with the work so auspiciously begun by Faraday, and with the liquefication and solidification of air and hydrogen by my predecessor in this chair this chapter in the history of physical science closes with the closing years of the century. It will ever be accounted among the triumphs of the 19th century that it has effected the removal of those artificial boundaries—the hard and fast lines of demarcation—implied by terms which have now no other rational meaning than as denoting states of physical aggregation. Nor will it be forgotten that it is largely to the labours of our countrymen, Dalton, Faraday, Andrews, and Dewar, that this achievement is to be ascribed.

In 1825 Faraday discovered benzene. He found it among the products furnished by the operations of the Portable Gas Company. Although he ascertained that the density of its vapour was 39, hydrogen being 1, the

* Presidential Address to the Chemical Society, 1900.

significance of that circumstance was not perceived at the time. The work of Ampère and Gay Lussac, in fact, was not really understood or appreciated at this period, and the conception of Avogadro lay dormant. "With regard to the composition of this substance," wrote Faraday, "my experiments tend to prove it a binary compound of carbon and hydrogen, two proportionals of the former element being united to one of the latter." What Faraday's bicarburet of hydrogen has developed into need not now be told. The work which has accumulated round this single substance during the seventy-five years which have elapsed since it has been known constitutes one of the most astonishing records of intellectual and industrial activity of which history has any record.

Faraday's next contribution to aromatic chemistry consisted in a study of the action of sulphuric acid on naphthalene, or "naphthaline" as it was then termed—a name which we owe to Dr. John Kidd, who lectured on chemistry at Oxford in the early part of the century, and who accurately described the main properties of the hydrocarbon, which appears to have been first observed in 1819 by Alexander Garden, of Old Compton Street, Soho, where the London Chemical Society had its habitation. The products obtained by Faraday are now known as α- and β-naphthalenesulphonic acids, the salts of which he described. It is noteworthy that in this year (1826) the Friday evening discourses at the Royal Institution were instituted by Faraday, with a view, as he says, to facilitate our object of attracting the world and making ourselves, with science, "attractive to it," and that this work on naphthalenesulphonic acid was the subject of the first chemical discourse of the series. Unpromising as it might seem, we may be quite sure that Faraday made it attractive.

Although Faraday continued to occupy himself at times with chemical subjects, even to the end of his tenure of the Fullerian professorship at the Royal Institution, he gradually became more and more absorbed in those memorable physical researches with which his name will ever be associated. The more important portion of his chemical work was practically all done before the end of 1826, and, curiously enough, one of the last inquiries with which he concerned himself in that year serves to connect him with Graham, whose services to science began in that year. It happened that Faraday, in 1823, had made some observations, perhaps among the earliest ever made, which served to establish what we now know as the transpiration of gases, but their accuracy had been doubted by Davy, who, three years later, repeated them. Davy's experiments on this subject constitute, in fact, the last ever performed by him in the laboratory of the Royal Institution. Faraday independently repeated the observations of 1823, and established their validity.

Graham's chief work, of course, lay in the borderland between physics and chemistry. The finite extent of the atmosphere; the absorption of vapours by liquids; the law of the diffusion of gases; the motion of gases; the diffusion of liquids; osmotic force; on liquid transpiration; on molecular mobility of gases; the absorption and dialytic separation of gases by colloid septa; on occlusion of gases; hydrogenium—however different may be the titles of his papers, all his work centred round conceptions of atoms and molecules and their motions. Of his contributions to pure chemistry, the most important was that "On the Arseniates, Phosphates, and Modifications of Phosphoric Acid"—the first of his papers sent to the Royal Society, to which it was communicated by Edward Turner, whom he succeeded a few years later in the Chair of Chemistry at University College. This memoir established the existence of three modifications of phosphoric acid—ortho-, pyro-, and meta-phosphoric acid. The first, or ordinary phosphoric acid, has been known for at least three centuries; pyrophosphoric acid was discovered by Clark, whom Graham succeeded as Lecturer on Chemistry at the Mechanics' Institute, Glasgow; metaphosphoric acid was now made known for the first time. The paper is memorable from the circumstance that it first definitely indicated the idea of *basicity*.

Graham's memoirs on the part played by water as a constituent of salts, and on the constitution of salts, are logically connected with and dependent on this paper. Indeed, nothing is more remarkable in Graham's work than the continuity of idea which pervades it. Just as the long succession of memoirs on the molecular movements of gases and liquids gave a definiteness and precision to the conception of the intestinal motions in matter, so his work on acids and salts served to establish the constitutional analogy between these substances.

Edward Turner was born in Jamaica in 1798, and, after studying medicine at Edinburgh, passed over to Göttingen, where he worked under Stromeyer. In 1824 he became a lecturer in Edinburgh, and here published an "Introduction to the Study and Laws of Chemical Combination," which he subsequently worked into his excellent "Elements of Chemistry," one of the standard text-books of the time, and by which he is mainly remembered. In 1828 he was made Professor of Chemistry in the newly-created University of London, now known as University College, and died in 1837 in the fortieth year of his age. Among his pupils was the late Sir Richard Quain, who described him as a lucid lecturer and a good analyst. Turner was indeed an excellent manipulator, and his analytical and determinative work was of a high order. He is especially to be remembered for his determinations of the atomic weights of lead, chlorine, silver, barium, mercury, nitrogen, and sulphur; they were the first atomic weights to be accurately ascertained by a British chemist, and are worthy to be ranked with the determinations of Berzelius. Turner's numbers were not only valuable as constants, but they were of theoretical importance as directly impugning the validity of Prout's hypothesis, which, mainly on the authority of Thomas Thomson, who had sought to support it by what is perhaps the weakest experimental evidence ever put forward, was part of the current doctrine of the time in this country. On Thomson's work Berzelius had passed what is probably the most scathing criticism he ever uttered:—"This work belongs to those few productions from which science will derive no advantage whatever. Much of the experimental part, even of the fundamental experiments, appears to have been made at the writing-desk; and the greatest civility which his contemporaries can show its author is to forget that it was ever published" (*Jahresber.*, 1827, 77).

Turner pointed out (1) that the atomic weights hitherto commonly used by British chemists had been adopted without due inquiry, and that several of the most important ones were erroneous, and (2) that the hypothesis that all equivalents are multiples of a whole number of the equivalent of hydrogen was inconsistent with the state of chemical knowledge at the time, being at variance with experiment. "Under such circumstances Dr. Prout's hypothesis, as advocated by Dr. Thomson—that all atomic weights are simple multiples of that of hydrogen—can no longer be maintained. I grant most willingly that a system of whole numbers, considered as moderate approximations, may, with advantage, be retained for the use of medical men, students, and manufacturers; but as the strict representative of scientific truth, applicable to all the purposes of science, this hypothesis is at present untenable. Let me not, however, be misunderstood: I mean simply to affirm that the experiments by which it has been attempted to prove the truth of this hypothesis are inaccurate; I may go further, and declare it to be not only unsupported by evidence, but to be at variance with the most exact analytic researches which have been conducted. I deny not that some simple relation subsists among atomic weights, and that their ratios may possibly be expressed by some series of numbers; but at present no one has assigned any physical cause for the existence of such a relation; no such relation has hitherto been discovered; nor, as appears to me, has analytical che-

mistry attained that degree of perfection which can justify anyone in finally asserting or denying its existence." The position thus taken up by Turner is precisely that to which Stas arrived half a century later, after an inquiry which will ever remain a model of analytical skill.

William Hyde Wollaston died on December 22nd, 1828, at the age of sixty-two; Thomas Young on May 10th, 1829, aged fifty-six; and Humphry Davy on May 29th, 1829, aged fifty-one. The passing away of three such men in the ripeness of their intellectual vigour, and in such quick succession, profoundly affected the whole scientific world. They had been colleagues during life, each holding high office in the Councils of the Royal Society, and were, with Dalton, the brightest ornaments of one of the most glorious epochs in the history of physical science in this country. At present we are only concerned with Wollaston and Davy. On Davy's triumphs, on his mental powers, and on the characteristics of his genius, it is unnecessary now to dwell; with the exception of that of Dalton, there is probably no personality in the history of British chemistry with which we are more familiar. Wollaston is less well known; his achievements, indeed, were less calculated to strike the popular imagination than those of his great contemporaries. But this silent, austere man, who lived in and for his laboratory and his library, his only relaxation being an occasional evening among his fellows at the Royal Society Club, or at the gatherings of the Royal Society, had a wonderfully clear intelligence and an astonishingly penetrative insight. No man ever more justly earned the title of philosopher. He was not so prolific or so diffuse as Priestley, but his learning was infinitely deeper and broader, and there is not a single branch of the science of his day which he did not illumine and adorn,—chemistry, astronomy, optics, mechanics, acoustics, mineralogy, crystallography, botany, physiology, pathology.—each in turn was the subject of his intellectual activity. It is, however, only with his chemical work that we have here to do. Reference has already been made to the part which Wollaston's paper, "On Super-acid and Sub-acid Salts," played in securing attention to the atomic theory; his "Synoptical scale of chemical equivalents" was of special service to the student and the manufacturer in facilitating the application of the laws of chemical combination to operative chemistry. His "Description of a reflective Goniometer" is a classic in the literature of chemical crystallography, and marks an epoch in the history of that subject. His discovery of a process by which platina may be rendered malleable proved of incalculable service to practical chemistry and the arts. Some idea of what that service has been may be gained by recalling what we have secured, and secured only by our crucibles and other vessels of platinum, and by the use of platinum foil and wire, and by imagining what our position would be if we were wholly deprived of these articles. Platinum had been known for some sixty years before Wollaston's time, but, as has been stated, "the very properties which made its value certain if it were wrought into vessels, forbade its being easily fashioned into them," and whole cargoes of the native metal are said to have lain unpurchased for years in London, as it could not be turned to account. Wollaston, foiled in his attempts to make a living by medical practice, became, as is well known, rich by his process of working platinum, which he carried on in secret and with the aid of a faithful old servant in his laboratory, a small detached building at the bottom of his garden, in Hunter Street, Brunswick Square.

In the early part of the century the price of manufactured platinum was less than half its present value. In 1805 platinum crucibles were to be obtained from Mr. Carey, 182, Strand, at 17s. 6d. the ounce, whilst wire was 16s. At the time of the foundation of the Chemical Society, the price of manufactured platinum had more than doubled.

Wollaston first made known his process in the Bakerian Lecture of 1828—the year of his death—and the Council of the Royal Society "deemed themselves bound to express their strong approbation of this interesting memoir by awarding a Royal medal to its author, and they anticipate with confidence a general approbation of what they have done." The further development of the metallurgy of platinum in this country is associated with the firm of Johnson, Matthey, and Co., who worked platinum for many years before any other commercial manufactory was established. The process of Wollaston appears to have been first worked, sometime between 1800 and 1808, by Mr. Thomas Cock, a relative of Mr. Percival Johnson, who began the refining and manufacture of platinum in Hatton Garden.

Wollaston's name is further connected with the platinum group of metals by his discovery of rhodium, which he published in 1804, immediately after the existence of iridium and osmium had been announced by Smithson Tennant, and also by his discovery of palladium. The manner in which the last-named metal was made known constitutes one of the most singular episodes in the history of scientific discovery, and affords a curious commentary on certain phases of Wollaston's character. In fact, one or two points in Wollaston's connection with the discovery and working of the platinum metals are somewhat obscure.

In 1803 appeared an anonymous circular which was sent to a number of persons, amongst them the editors of *Nicholson's* and *Tilloch's Journals*, stating that a new metal—palladium or new silver—could be purchased from Mrs. Forster, 26, Gerrard Street, Soho, in samples of 5s., half-a-guinea, and 1 guinea each, the price being at the rate of 1s. per grain. Certain of the properties of palladium were given in the advertisement to show that it was "a new noble metal." This announcement attracted the attention of Chenevix, an Irish gentleman, well known as a Fellow of the Royal Society, and for his analyses of certain minerals and for his work on oxygenised and hyperoxygenised muriatic acid. The mode adopted to make known a discovery of so much importance, without the name of any creditable person except the vendor, appeared to Mr. Chenevix unusual in science and not calculated to inspire confidence. Accordingly, with a view to detect what he conceived to be an imposition, Mr. Chenevix procured a specimen and eventually the whole quantity which had been left for sale with Mrs. Forster, who, she stated, was totally unacquainted with the person who brought the metallic substance and the printed paper to her house. Chenevix found all the statements in the paper to be correct with the exception of that of the specific gravity, which he stated to be different by 1 or 2 per cent. He made a long and elaborate investigation of the matter, and concluded that the pretended new metal was a combination of platinum and mercury. He sent in his memoir to the Royal Society, and two evenings were spent in the reading of it.

The Journal Book of the Society, the entries of which were made under the direction of the Secretaries, if not actually by them, gives a *précis* of this paper. We read: "Thus, after having been baffled in his attempts to discover, by analysis, the component parts of this substance, which he (the author) could never bring himself to consider as a new metal, a synthetic process at length led him to the discovery that the whole pretence was an imposition, and that the substance is, in fact, a combination of platina and mercury; in which the latter, while it masks the most characteristic properties in the former, loses the greater number of its own distinctive qualities.

"The singular fact that an alloy of two metals should be produced, the specific gravity of which is little more than one-half of what it ought to be by calculation, is, no doubt, worthy of particular attention; and as quicksilver was in this process brought to a fixed state under circumstances never before observed, a notion might be entertained that the great desideratum in alchemy, the fixation of mercury, was by no means a visionary object. . . . Those who cultivate chemistry with any degree of ardour

will be gratified to see in this paper the pains taken by the author, and the various modes he has devised, to produce this compound metal in its most perfect state of combination."

A few months later, the editors of the various scientific periodicals received "under cover of the twopenny post," an unsigned paper offering a reward of £20 for the artificial production of palladium. The editor or *Nicholson's Journal* says the paper "is written in the same hand as a note which covered a small piece of palladium mentioned to have been received by me last midsummer. Upon inquiry, I find that Mrs. Forster has received the sum of £20 with instructions conformable to this paper. The paper ran as follows:—

December 16, 1803.

"Sir,—As I see it said in one of your journals, that the new metal I have called palladium, is not a new noble metal, as I have said it is, but an imposition and a compound of platina and quicksilver, I hope you will do me justice in your next, and tell your readers I promise a reward of 20£ now in Mrs. Forster's hands, to any one that will make only 20 grains of real palladium, before any three gentlemen chymists you please to name, yourself one if you like.

"That he may have plenty of his ingredients, let him use 20 times as much quicksilver, 20 times as much platina, and in short of anything else he pleases to use: neither he nor I can make a single grain.

"Pray be careful in trying what it is he makes, for the mistake must happen by not trying it rightly.

"My reason for not saying where it was found, was, that I might make some advantage of it, as I have right to do.

"If you think fit to publish this, I beg you to give the names of the umpires, as I have desired Mrs. Forster to keep the money till next midsummer, and to deliver it only in case they can assure her that the real metal is made by a certificate signed by you, and by them, on this check.

"I hope a little bit of whatever is made may be left with Mrs. Forster."

Mr. Nicholson nominated Hatchett and Edward Howard to join himself as judges of the product which might be made in their presence; but the £20 was never claimed, and in 1805, Wollaston, in a memoir contributed to the *Philosophical Transactions*, announced that he was the discoverer of the new metal, to which he had given the name palladium, "from the planet which had been discovered, nearly at the same time, by Dr. Olbers."

What was Wollaston's motive in bringing his discovery to the notice of the scientific world in so extraordinary a manner can only be surmised. From the account of his memoir given in the Journal Book of the Society, in which no mention is made of Chenevix and his work, he refers to the "concise delineation of its character" given in the advertisement of the metal, in which he says he "avoided directing the attention of chemists to the source from where it had been obtained, and thereby reserved to himself a more deliberate examination of many phenomena that yet remained unexplained in the analysis of platina, by which he was subsequently led to the discovery of rhodium." No proper explanation was given of the circumstance that he, as Secretary of the Society, allowed Chenevix's paper to appear in the *Transactions*. It must have been his duty actually to read this paper at the two meetings when it was brought before the Fellows; he must also have been responsible for the account which appears in the Journal Book, and which, as one now peruses it, in the light of what subsequently happened, is almost cutting in its sardonic humour and irony. It has been stated that Wollaston did all in his power to persuade Chenevix to withhold or withdraw his paper. Be this as it may, there is no doubt that Chenevix keenly felt the humiliation he suffered. He retired to the Continent, and all his subsequent papers were published in France.

(To be continued).

NOTICES OF BOOKS.

Calculations on Analytical Chemistry. By Edmund H. Miller, Ph.D. London and New York: Macmillan and Co., Ltd.

Dr. Miller, of Columbia University, has written a text-book for the use of students in schools and colleges. His aim has been to explain clearly the principles involved in making the various calculations required in analytical chemistry, and we think that no one who has thoroughly grasped the principles as set forth and illustrated in this work should be at a loss in performing similar calculations however complex the material with which they are dealing.

The work consists of ten chapters and a score of tables. Chapter I. explains the method of obtaining the chemical equivalent of an element and how from this the atomic weight may be determined. The following seven chapters deal with the methods of analysis of compounds and mixtures in the three states, whilst the ninth is devoted to the consideration of the heat absorbed or set free by chemical reactions, and the last chapter deals with electro-chemical equivalents.

More than 200 examples are given throughout the work; many are fully worked out in the body of the work, the remainder being placed at the ends of the chapters to enable the student to test his knowledge. We have verified many of the examples, and the only error we could detect was on p. 126, line 14, where $\frac{20000}{10}$ should be $\frac{20000}{10-1}$.

History of Chemistry, since the time of Lavoisier. By Dr. A. Ladenburg. Translated from the second German Edition by L. Dobbin, Ph.D. With additions and corrections by the author. Edinburgh: The Alembic Club, William F. Clay. 1900. Pp. 373.

The science of chemistry assumed a new aspect in the hands of Lavoisier, and it is for this reason that the author has not gone back further for the starting point of this history.

Before the time of Lavoisier the "phlogiston" theory was the accepted one of chemists, but shortly after the discovery of oxygen, Lavoisier repeated the experiments of Priestley and Scheele, but drew conclusions from them totally different to those of the two other chemists. He at once recognised oxygen as that part of the air which unites with the combustible matter during its combustion, and some time afterwards, in the year 1777, he advanced a complete theory of combustion in which he says:—"1. Heat annd light are disengaged in every combustion. 2. Substances burn only in pure air. 3. This is used up in the combustion, and the increase in weight of the substance burned is equal to the loss in weight of the air. 4. The combustible substance, by its combination with pure air, is usually converted into an acid; the metals, on the other hand, are converted into metallic calces."

Lavoisier adheres to Boyle's definition of an element, and this definition is retained to the present day. He, however, not only overthrew the old "phlogiston" theory, but he introduced a new one in its place, the principal points in which are his statement, that in all kinds of chemical reactions it is the kind of matter alone that is changed, whilst its quantity remains constant; that all acids contain oxygen united, as he expresses it, with a basis or a radical, and the combustion theory referred to above.

We have dwelt rather long on Lavoisier's work as it marks an important epoch in the development of modern chemistry. Many other writers and thinkers have followed him and gradually brought the science up to its present form. Dalton's atomic theory was another great advance, as also was the inception and development of the radical and the nucleus theories.

All this and much more is fully discussed and analysed in these interesting pages. Chapter XVI., the last, has been specially written for this English edition, and treats of the development of chemistry during the last fifteen or twenty years. This latter development is distinguished by the increasing prominence of physical or general chemistry. To this end such workers as Horstmann, Gibbs, Van der Waals, and van 't Hoff have largely contributed.

Again, Moissan on the one hand, and Dewar on the other, have achieved great scientific results, the one in his work on very high temperatures, and the other in the production of extreme cold.

Our knowledge of the composition of the atmosphere has also been much extended owing to the labours of Lord Rayleigh and Professor Ramsay, while the development of the chemistry of the carbon compounds has advanced to an unexpected extent.

What the coming phases of the science may be, nor how our present ideas will appear to future investigators, none can foretell; but as a history of what chemistry has been during the last 130 years, this book is of great service, and deserves the success it has achieved.

Laboratory Note-book for Chemical Students. By Vivian B. Lewes and J. S. S. Brame. London: Archibald Constable and Co. Pp. 170.

We are glad to come across a laboratory note-book such as this, which has been compiled, not with reference to any particular syllabus or examination, but to cover such practical work as a student should be familiar with if he is to have a good ground-work in the subject of practical chemistry. By omitting long descriptions of such simple matters as filtration, weighing, &c., which often fill up many pages, the authors have been enabled to include all the principal reactions of the metallic and acid radicals, besides the preparation of gases, &c., and some of the more simple quantitative experiments. In Part II. we find the usual tables given for the group separation of the bases, and a general scheme for the analysis of mixtures. Part III. is devoted entirely to experiments of a quantitative character, and includes the simple examination of water, fuels, lubricating oils, and explosives, these latter being more especially needed by students desirous of entering the army or navy. The book is plentifully interleaved with blank sheets for the student's own notes, working out equations, &c., the whole making a welcome addition to this class of literature.

The Measurement of High Temperatures. ("Mesure des Températures élevées"). By H. Le Chatelier and O. Boudouard. Paris: G. Carré and C. Naud. 1900. Pp. 220.

The first to take up the measurement of high temperatures was Wedgwood, the celebrated Staffordshire potter, and in the year 1782 he constructed and used a pyrometer in which he utilised the contraction of clay; this pyrometer was the only one in use for nearly a century.

Since Wedgwood's time many savants have been engaged in the study of the measurement of high temperatures with varying success. As a few examples of the kinds of thermometers used for high temperatures we may mention the following:—Wedgwood's, which registers by the permanent contraction of clay; Pouillet's, by the dilatation of air; and Siemens's, by the alteration of electric resistance of platinum.

The authors first consider the normal scale of temperatures, that is the thermo-dynamic scale; but as it is impossible to utilise this with accuracy, it becomes necessary to have a practical scale, in a similar manner to the actual metre rod which is kept as a standard at the Bureau International des Poids et Mesures. This practical scale exists as a hydrogen gas thermometer of constant volume charged with hydrogen at a pressure of 1000 m.m. of mercury at the temperature of melting ice, but with high temperatures it is not possible to get closer results than 1°, and practically even greater differences occur.

Electric pyrometers have enabled us to determine a large number of temperature points, concerning which our ideas were very vague, and it is with this instrument that M. Osmond, Sir William Roberts-Austen, M. Charpy, and others have carried out their researches on the molecular transformations of irons and steels.

Pyrometers for measuring calorific radiations are also described, as well as those by which high temperatures may be estimated by means of luminous radiations: these are similar to those last mentioned, with this difference that they only use the visible rays, and not the invisible or heat rays; it is by these means that the highest temperatures of all can be measured.

The authors have handled the subject with great ability, and have produced a really excellent work on this subject.

The Photographer's Note-book and Index. By Sir David Salomons, Bart., M.A. London: Marion and Co.

This useful note-book has been revised, and much new matter added. It contains a lot of information and rules in a compact form, for taking, printing, and enlarging photographs, &c., and a number of ruled pages for entering the details of conditions under which the photographs were taken, such as length of exposure, developer used, &c.

A Treatise on Crystallography. By W. J. Lewis, M.A. Professor of Mineralogy in the University of Cambridge. Cambridge: The University Press. 1899. Pp. 612.

The author has set out clearly in this volume the views held at the present time as to the classification of crystals and the principles of symmetry on which the classification is based; his treatment of the subject has been as far as possible geometrical, and, with the exception of the formulæ of plane and spherical trigonometry necessary for the solution of triangles, very little analysis has been introduced into the main discussion.

By the principles laid down and the relations established in the earlier chapters the classification of crystals is rendered easy, and it is shown that there are only thirty-two classes possible, and these classes are arranged in seven larger groups known as systems. It is now generally held that each of the thirty-two possible classes of crystals is a definite group, the forms of which are a direct consequence of the elements of symmetry present in it. The names adopted by various authors for the systems and classes differ considerably; Professor Lewis has generally adopted Miller's names for the systems, but he also gives some of the synonyms employed by other crystallographers. The systems and the classes included in each system are fully developed in Chapters XI. to XVII.; these are followed by a chapters on twins and other composite crystals.

Twin crystals can be divided into two main classes according to the manner in which the parts are united; these are: 1. Juxtaposed twins in which the portions are united along a plane surface, and lie on opposite sides of it; and 2. Interpenetrant twins in which the portions are intimately mingled without any regular surface of separation between their matter. Chapter XX., and last, deals with the goniometer, the different forms of which are fully described and illustrated.

The diagrams in a work of this class are of considerable importance, as good figures are great aids to the understanding of the geometrical relations of crystals. We

must congratulate the author on the excellence of the diagrams, which appear throughout to have been most carefully prepared.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. CXXX., No. 17, April 23, 1900.

Heat of Combustion and Formation of Iodine Compounds.—M. Berthelot.—The author employs the calorimetric bomb and compressed oxygen to determine the heat of combustion and heat of formation of iodine compounds. He finds that the iodine is liberated without any sensible formation of hydriodic or iodic acids. Such compounds as iodoform, CHI_3, and periodoethylene, C_2I_4, which are rich in iodine, burn easily and entirely in compressed oxygen. By this method he is able to employ considerable quantities of the compound, even as much as 8 grms. in certain cases, and so ensure precision of the determinations. Numbers were found for methyliodhydric ether, CH_2I, methylene iodide, CH_2I_2, iodoform, CHI_3, ethyliodohydric ether, C_2H_5I, normal propyliodohydric ether, C_3H_7I, isopropyl iodide, C_3H_7I, allyl iodide, C_3H_5I, iodobenzine, C_6H_5I, iodobenzoic acid, $C_7H_5IO_2$, iodosalicylic acid, $C_7H_5IO_3$, di-iodosalicylic acid, $C_7H_4I_2O_3$, ethylene iodide, $C_2H_4I_2$, di-iodoform and iodol, C_4HI_4N.

New Indicator for Acidimetry and its Application to the Estimation of Boric Acid.—Jules Wolff.—The author uses as an indicator ferric salicylate in a solution of sodium salicylate. A solution of boric acid added to a given volume of sulphuric acid of known strength coloured with the new indicator, changes colour at the precise moment of saturation of the sulphuric acid by the standard soda solution, from violet to orange, the latter tint persisting in presence of excess of alkali. When the sulphuric acid is in great excess, the addition of the indicator at first produces no colouration, but the colour comes as the neutralisation of the acid proceeds. The author obtains the indicator in a crystalline state. He finds that it is very sensitive in presence of alkaline bases, also in the presence of sulphuric, nitric, hydrobromic, hydriodic, and hydrochloric acids. In presence of phosphoric and hydrofluoric acids the violet colour is not produced.

Selenides and Chloro-selenides of Lead.—M. Fonzes-Diacon.—The author prepares crystalline selenide of lead (1) by the reduction of the selenate under the influence of hydrogen or carbon; (2) by the action of hydrogen selenide on the vapour of lead chloride; and (3) by direct fusion, in the electric furnace, of precipitated lead selenide. He was unable to prepare the sub-selenide, but succeeded in preparing, both by the wet and dry method, a chloro-selenide of lead.

The Alkaline Selenio-antimenites.—M. Pouget.—Little is known of the compounds of antimony selenide with the alkaline selenides. Two compounds only have been described:—$SbSe_4Na_3+9H_2O$ and $SbS_3SeNa_3+9H_2O$. The author succeeds in preparing several other selenio-antimonites by a method identical with that used for the preparation of alkaline sulphantimonites, *i.e.*, by the action of antimony selenide on dissolved alkaline selenides. Among these are $Sb_2Se_3+3K_2Se$, $SbSe_3Na_3+9H_2O$, $Sb_2S_3K_4+2SbSe_3K_3+4H_2O$.

Micro-chemical Researches on Yttrium, Erbium, and Didymium.—E. Pozzi-Escot and H. C. Couquet.—The following reactions allow of the identification of yttrium, erbium, and didymium:—(1) Salts of yttrium give, with chromate of ammonium, an yttria chromate which crystallises, after some minutes, in the form of voluminous hexagonal elongated crystals, slightly tinted violet. (2) Yttrium salts give, with a solution of chromic acid in concentrated sulphuric acid, small crystals, which, after a time, become transformed into voluminous crystals (Couquet's reaction). (3) Erbium salts give, with ammonium chromate, a compound which either does not crystallise at all, or else does so with great difficulty. (4) Erbium salts give, with Couquet's reaction, large badly-defined crystals. (5) Didymium salts give, with ammonium chromate, a didymium chromate formed in large orange prismatic crystals. (6) Didymium salts give, with Couquet's reaction, an amorphous precipitate, which, after some time, crystallises in prisms often split in the form of stars. (7) Chlorides of yttrium, didymium, and erbium give, with palladium chloride, characteristic double chlorides.

No. 18, April 30, 1900.

Manganous Fluoride.—Henri Moissan and M. Venturi.—Manganous fluoride, MnF_2, can be easily prepared in the state of a crystalline sand by the action of hydrofluoric acid on manganese. This substance may also be obtained in beautiful crystals by utilising the property it possesses of dissolving in melted manganese chloride. Manganous fluoride is insoluble in water, alcohol, and ether, so differing entirely from the chloride. It is a compound easily reduced both by metals and non-metals, giving, in many cases, crystalline products as a result of the decomposition. It is easy to add fluorine to the compound, and so prepare the crystalline sesquifluoride of manganese, MnF_6.

Samarium.—Eug. Demarçay.—Samarium prepared by the crystallisation of the double magnesium nitrites, as already described by the author, can be obtained in a state of comparative purity, showing after continuous fractionation the properties of the first and last portions to be absolutely identical, both with regard to the absorption and the spark spectrum. The absorption spectrum obtained by the author coincides in almost all respects with that previously published by Lecoq de Boisbaudran. The line 614·4 is, however, quite absent in this specimen of pure samarium. The author obtained the atomic weight of his samarium by the synthesis of the sulphate from samaria and sulphuric acid, varying between 147·2 and 148. Nitrate of samarium and magnesium, $Sm(NO_3)_2\,3Mg(NO_3)_2.24H_2O$, is a pale yellow salt, crystallising in large rhombohedra and melting between 93·5° and 94·5°. The simple nitrate, $Sm(NO_3)_3.6H_2O$, is formed in large orange-yellow crystals melting at 78—79°, and is very hygroscopic.

Compounds of Metallic Iodides with Sulphurous Anhydride.—E. Péchard.—When sulphur dioxide is allowed to pass into a solution of potassium iodide, the latter acquires a yellow tint, becoming orange-coloured if the solution is concentrated. This colouration is not due to the liberation of iodine. Solid potassium iodide which has been carefully dried absorbs also large quantities of sulphur dioxide and becomes yellow. Both the solid and the solution lose the absorbed gas by the action of heat. The author finds, therefore, a dissociable compound of potassium iodide and sulphurous anhydride. He first determines the dissociation tensions at different temperatures between 0° and 30°. An analysis of the compound shows that it probably has the formula $SO_2.KI$. Other soluble iodides form analogous combinations, those of sodium, ammonium, barium, and calcium having been prepared.

Bromination by means of Aluminium Bromide.—Ch. Pouret.—By allowing aluminium bromide to act on the chlorine derivatives CCl_4, C_2Cl_4, C_2Cl_6, the corresponding bromine derivatives are formed, CBr_4, C_2Br_4, C_2Br_6. This operation takes place in a glass bulb or in sealed tubes; in the latter case the yield is as large as 90 or 92 per cent. By using powdered aluminium on the same chlorides in presence of bromine, the author obtained in all cases C_2Br_6.

Gases emitted from the Mont-Dore Springs.—F. Parmentier and A. Hurion.—After collecting the gas from the spring, the authors used potash solution, and found that from 100 c.c. about 0·5 c.c. remained unabsorbed. This residue showed negative properties. Analysis of the gas gives the following result:—

Carbonic acid	99·50
Nitrogen	0·49
Argon	0·01
	100·00

Action of Monochloracetic Ethers on Sodium Acetylacetone.—F. March.—Bischoff, making use of the property possessed by bodies having a group CH_2 placed between two CO groups, of reacting with the halogen compounds of saturated hydrocarbides, investigated the action of monochloracetic ether both on sodium acetylacetic ether and on sodium malonic ether. He obtained by this means the acetyl succinic ether and the ethenyltricarballylic ether. Haller and Barthe, by allowing monochloracetic ether to react on sodium cyanacetic ether, prepared a mixture of cyanosuccinic and cyanotricarballylic ethers. The author finds that sodium acetylacetone reacts in the same way with monochloracetic ether.

Action of Ethylidene Chloride on Phenols.—R. Fosse and J. Ettlinger.—The authors investigate the action of ethylidene chloride on acetal of orthocresol, acetal of paracresol, and resorcine.

Presence of Tyrosine in Waters of Contaminated Wells.—H. Causse.—An examination of the water shows that when it is coloured yellow by chloromercurate of para-diazo-benzene sulphonate of sodium, tyrosine is present. The circumstance that the intensity of colouration increases with the disappearance of ammonia and the clearness of the water, shows that the tyrosine did not pre-exist, but appears to be due to an energetic oxidation which has a simultaneous effect on the mineral and organic substances present in the water.

MISCELLANEOUS.

Proposed University of Birmingham.—The report of the Advisory Committee as to the technical and commercial sections of the University of Birmingham has recently been issued. The committee have visited a number of Colleges and Universities, both at home and abroad, and have also had interviews with many of the leading manufacturers in the city and district, with a view to ascertaining the pecularities of the industries with which they are associated and the best means of meeting their needs, also the kind of technical knowledge desirable in the members of their staff. They have classified the industries of the district as follows:—1. Mining. 2. Metallurgy. 3. Engineering. 4. Chemical trades. 5. Non-metallic trades; and they recommend that there shall be chairs of Mining, Metallurgy, Engineering, and Applied Chemistry. The cost of the land, buildings, &c., for carrying out all the recommendations detailed in this report is estimated at £155,000, and the cost of maintenance (including the staff) at £10,450 per annum. A very important recommendation, which may not perhaps find favour with the staff, is that to properly carry out the necessary courses the usual vacations be considerably shortened, and the time of the students more fully occupied. The staff would also be expected to give their whole time to the service of the College, and should not be expected to assist their incomes by outside work. We are afraid that the salaries required by first-class men if such conditions were rigidly enforced would be prohibitive.

Merck's Digest, No. 7.—This number of the *Digest* contains an account of the two halogenised fats *iodipin* and *bromipin*. These products are obtained by the addition of iodine and bromine respectively to oil of sesame, or teel oil (from *Sesamum orientales*). They are yellowish liquids of a purely oily taste, and possess the physical properties of fatty oils. Iodipin is supplied commercially as a 10 per cent and a 25 per cent preparation, and bromipin is supplied at 10 per cent and 33⅓ per cent strengths, the latter in capsules. These two preparations have been the subject of comprehensive physiological and clinical experiments which show them to be efficient substitutes for the usual iodine and bromine preparations, especially the alkaline iodides and bromides, and free from their objectionable secondary properties.

The Preparation of Acrolein.—A. Wohl and L. Neuberg.—The author advantageously replaces the bisulphate of potassium, in the preparation of acrolein, with boric anhydride, or even with ordinary boric acid. To obtain the acrolein, 500 grms. of glycerin mixed with 335 grms. of crystallised boric acid are distilled in a metal retort of 3 litres capacity; this is first heated without the cover so long as steam comes off, and until the mass commences to turn black. It is then allowed to cool, the cover is then placed on the retort, which is heated strongly after having surrounded the apparatus with asbestos card. The distillation is finished after about three-quarters of an hour, and 154 grms. of acrolein are obtained, that is 50·5 per cent of theory. If the temperature is not sufficiently high at the commencement, the distillation will be too slow, and boric acid will be deposited in the cool part; this may cause disagreeable obstructions. The raw acrolein obtained in this manner is purified by distilling two or three times over chloride of calcium.—*Berichte*, xxxii., p. 1352.

MEETINGS FOR THE WEEK.

MONDAY, 28th.—Society of Arts, 4.30. "Imperial Telegraphic Communication," by Sir Edward Sassoon, M.P.

TUESDAY, 29th.—Royal Institution, 3. "Ruskin, Man and Prophet," by R. Warwick Bond, M.A.

WEDNESDAY, 30th.—Society of Arts, 8. "Russian Central Asia—Countries and Peoples," by A. R. Colquhoun.

THURSDAY, 31st.—Royal Institution, 3. "Chaucer," by the Rev. Canon Ainger, M.A., LL.D.

FRIDAY, June 1st.—Royal Institution, 9. "Bunsen," by Sir Henry Roscoe, F.R.S., &c.

SATURDAY, 2nd.—Royal Institution, 3. "The Growth of Chamber Music" (with Musical Illustrations), by Sir Frederick Bridge, Mus. Doc.

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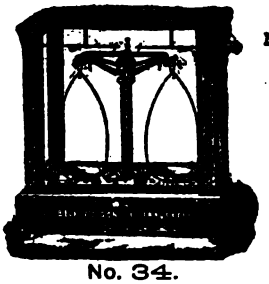

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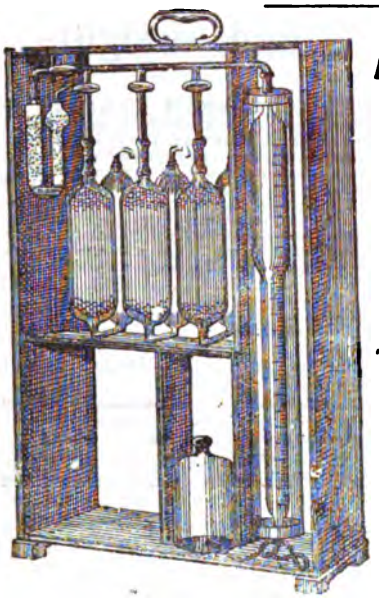

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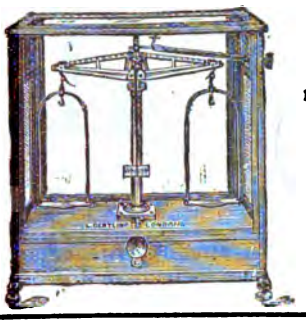

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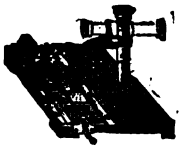

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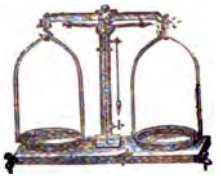

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THE CHEMICAL NEWS.

Vol. LXXXI., No. 2114.

RADIO-ACTIVITY OF URANIUM.*

By Sir WILLIAM CROOKES, F.R.S.

1. The researches of M. Henri Becquerel have shown that compounds of uranium possess the property now called radio-activity; that is, rays emitted by them affect a sensitive photographic plate through bodies usually considered opaque to light; they discharge an electrometer when brought near it; and they are deflected by a magnet. These rays are now called "Becquerel rays," or "uranic rays."

2. On the discovery by M. and Mdme. Curie of polonium and radium, bodies of enormous radio-active powers, it was suggested that uranium might possibly owe its power to the presence of a small quantity of one of these bodies. But in a paper published in the *Revue Générale des Sciences* for January, 1899, Mdme. Curie says:—"This does not appear probable, for if such were the case different samples of uranium compounds would have very different radio-activities, but in the course of a number of experiments made with various samples of metallic uranium, as well as with oxides and salts from various sources, I have never found any marked difference between the relative activities of the same compound."

In another paper (M. and Mdme. Curie, *Comptes Rendus*, cxxvii., p. 175; Chem. News, lxxviii., p. 49, July 29, 1898), the same author says that "the property of emitting rays . . . . which act on photographic plates is *a specific property of uranium and thorium*." "The physical condition of the metal seems to be of an altogether secondary importance." "Uranium and thorium alone are practically active."

3. When the discovery of radium was announced, and it was said to have "to all appearance the properties of almost pure barium" (M. and Mdme. Curie and M. Bémont, *Comptes Rendus*, cxxvii., p. 1215; Chem. News, lxxix., p. 1, January 3, 1899), it occurred to me that radium might be found in detectable quantities in some barium minerals were search made among them from different localities. Accordingly specimens of the following minerals were put on sensitive plates, a sheet of black paper separating them from the sensitive surface. As it was probable that the radio-active substance would be present, if at all, in very minute quantities, the sensitive plate was exposed to their influence for forty-eight hours.

Barytes (Heavy Spar).
" from Hungary. (Three specimens).
" " Cumberland. (Eight).
" " Westmoreland. (One).
" " Cumberland. (A fine crystal).
" " Derbyshire. (Three).
" " Arkendale. (One).
" " Hartz. (One).
" " Scotland. (One).
" " Ireland. (Two).
" " Northumberland. (Two).
" " Arran. (One).
" " Cherbourg. (One).
" " Several unnamed, but finely crystallised, specimens.
Witherite from Lancashire. (One).
" " Cumberland. (Four).
" " Northumberland. (One).

Not one of these minerals showed the slightest action on the sensitive plate.

4. Having obtained negative results with barium compounds, I went through every mineral in my cabinet—a somewhat extensive collection, numbering many fine specimens. Large photographic plates were covered with black paper, and the minerals were laid on them as close as they could conveniently be placed, accurate note of their names and positions being recorded. They were exposed in total darkness for forty-eight hours. By this means a list of radio-active minerals was ultimately obtained. They were then tested for order of intensity of action. The following is a list of active minerals arranged in order, the most active heading the list:—

1. Pitchblende.
2. Uranite.
3. Autunite.
4. Orangite.
5. Thorite.
6. Euxenite.
7. Samarskite.
8. Alvite.
9. Broggerite.
10. Monazite.
11. Xenotime.
12. Arrhenite.
13. Sipilite.
14. Fergusonite.
15. Chalcolite.
16. Hielmite.

It will be observed that these minerals all contain either uranium or thorium.

5. Pitchblende was the most radio-active mineral, but it varied much in different parts. A slice was cut from a piece of pitchblende from Cornwall and the surface was polished. A sensitive photographic plate was pressed against it, and after twenty-four hours the plate was developed. The impression showed the structure of the mineral in a remarkable manner, every little piece of pitchblende showing black, those portions in which the radio-active substance was not so operative showing in half tint, while the felspar, quartz, pyrites, &c., having no radio-activity, left the plate transparent.

Pitchblende from different localities differed greatly in action.

6. A large crystal of orangite from Arendal was ground flat and polished at one end and side, and a piece of sensitive celluloid film, cut half through so as to allow it to bend sharply, was put on the polished surfaces, one half pressing against the end and the other half against the side. The exposure was continued for seventy-two hours. On developing, no difference could be seen in the intensity of the impression, whether made by the end or the side of the crystal. The impression also was uniform over the surface, the cracks in the surfaces not having impressed themselves.

These experiments were repeated with the interposition of a thin sheet of celluloid between the mineral and the sensitive plate. The results were practically the same as before.

7. Roughly speaking, the action of pitchblende is in proportion to the percentage of uranium in the mineral. Finely powdered pitchblende of different degrees of richness were experimented with. Cells were made of thick lead pipes half an inch internal diameter and one inch long, closed at the lower ends with card. They were filled with powdered pitchblende, one containing 43 per cent Ur_3O_8 and the other 12 per cent Ur_3O_8. A sensitive plate being covered with black paper, the lead cells were laid on it and kept in total darkness for 120 hours. The intensity of the spot under the 43 per cent ore on development was found to be at least three times that of the one under the 12 per cent ore.

Two lead cells were taken, one being a quarter of an

* A Paper read before the Royal Society, May 10, 1900.

inch long and the other two inches long. They were filled completely with the 43 per cent ore, and a sensitive plate exposed to their action for forty-eight hours. On developing it was doubtful whether any difference existed in the intensity of the two spots, proving that the action does not pass through much thickness of active material, a quarter of an inch being equal in effect to two inches.

No difference in the action was noticed when the bottom of the cell was made of thin glass cemented on, instead of card.

Four cells were filled with pitchblende and placed side by side on a sensitive plate. After having acted twenty-four hours the first was removed, the second after forty-eight hours, the third after seventy-two hours, and the last was kept on for ninety-six hours. On developing the plate the spots had intensities varying with the lengths of exposure, and in about the right proportion, on the assumption that double the time of action gives double the intensity of blackening.

8. For convenience of comparison I had a number of glass cells made, three quarters of an inch wide and deep, so that they could either be sealed up or closed with a cork. A piece of apparatus was made so as to take radiographs of samples with more ease and certainty. A lead plate, 2 m.m. thick, 6½ inches long, and 2½ inch wide, has circular holes punched in it, one inch in diameter. Under the thick plate of lead is another thinner plate, made of pure assay foil, and having holes in it, concentric with the others, but barely ¾ inch in diameter, so that one of the small cells will not pass through the lower hole, but will pass easily through the upper hole, and thus be kept in place. To prevent contact between the lead plate and the sensitive surface a thin sheet of celluloid is fixed beneath, with holes punched in it concentric with those in the lead plates. In the top left corner of the lead plates is a short steel pin, which can be pressed on the sensitive plate and so register its position in respect to the cells experimented with. The lead and celluloid plates are then bound together and the whole is fitted into a shallow wooden tray with a light-tight cover.

A sensitive film is laid face upwards at the bottom of the wooden tray; on this are put the lead screens, and then the experimental cells of radio-active bodies in order, careful note being taken of their relative positions.

9. Wishing to prepare compounds of radium and polonium, the very curious bodies discovered by M. and Mdme. Curie, I arranged with my friend Mr. Tyrer, Stirling Chemical Works, for the systematic working up of half a ton of pitchblende. It was necessary to examine every precipitate and filtrate in each stage of the operation, and for convenience of registration they had to be compared with a standard cell filled with a substance unvarying in action. After many trials, I selected crystallised uranium nitrate as being strongly radio-active and easily prepared pure. This led me to the observation which forms the subject of the present paper.

10. The following compounds of uranium were tested simultaneously, being put into glass cells and arranged on a lead screen with seven holes:—

1. Metallic uranium, from M. Moissan.
2. Uranium nitrate, $UO_2(NO_3)_2.6H_2O$.
3. Uranium acetate.
4. Uranium persulphate.
5. Uranium protosulphate.
6. Uranium oxide (green), $UO_2 2UO_3$.
7. Uranium oxide (black), UO_2UO_3.

For twenty-four hours the sensitive plate was exposed to the influence of these bodies. With the exception of metallic uranium, which showed least action, there was not much difference between the effect produced by any of the others.

11. In order to prepare uranium nitrate of great purity for a standard, I took some pounds of the commercial salt and purified it, first by solution in ether (13), and then by repeated crystallisation. After many operations a cell was filled with the crystals, and it was used as a standard. To my surprise, after having acted on a sensitive plate for twenty-four hours, on development not a trace of image could be seen.

12. Thereupon I tried the following experiments to ascertain if the great radio-activity of some uranium compounds and the absence of it in others might be caused by some variation of physical, crystalline, or chemical condition.

Commercial uranium nitrate was taken, and—

(1) A portion was heated with excess of nitric acid to dryness on the water-bath. It was powdered and put in a cell.

(2) A similar portion of the salt was dissolved in alcohol, and the solution evaporated to dryness over the water-bath. The resulting orange-coloured pasty material was ground and put in a cell.

(3) The salt was treated in the same way as No. 1, except that the excess of water and acid were not driven off.

(4) A small quantity was heated for some time on a water-bath till it was thoroughly dry. It was then powdered and put in a cell.

(5) 50 grains were put in a glass cell and heated on a water-bath to about 75° C. till it had dissolved in its water of crystallisation.

(6) 50 grains in a glass cell were heated on a sand-bath to about 230° C. The water of crystallisation having been driven off the salt fused, and on cooling remained a hard, yellow, glassy mass.

(7) A similar quantity of the same salt was heated to a little above 230° C. till it commenced to decompose.

(8) A similar quantity was heated more strongly, till about half the nitrate had decomposed.

(9) The same quantity was heated till decomposition was complete.

(10) As a standard, some of the same lot of commercial uranium nitrate from which these lots were taken was put in a cell.

These ten cells were placed in a lead screen apparatus, and a sensitive plate was exposed to their influence for twenty-four hours. On development there was not much difference between any of the impressions, that under No. 9 being a little the strongest.

Thus it appears that no modification of physical or chemical condition materially affects the radio-active property of a uranium compound when, to begin with, the salt experimented on possesses it; other similar experiments show that, starting with an inactive uranium salt, nothing that can be done to it will cause it to acquire this property. It is therefore evident that, as I had suspected, the radio-active property ascribed to uranium and its compounds is not an inherent property of the element, but resides in some outside body which can be separated from it.

Having by repeated crystallisation succeeded in preparing a photographically inactive uranium nitrate, I started experiments with several pounds of the commercial salt to ascertain the readiest means of separating from it the active body.

13. Into a stoppered cylinder I put 1 lb. of crystallised nitrate and poured on it a pound of methylated ether, sp. gr. 0·72. The salt easily dissolved on shaking, and after a few hours the whole of the crystals had disappeared, leaving at the bottom of the cylinder 1000 fluid grains of a heavy aqueous solution. I separated the aqueous solution from the ethereal solution, and evaporated it to dryness to remove traces of ether. The ethereal solution was allowed to evaporate spontaneously. Equal quantities of the soluble and the insoluble in ether I put into glass cells and added sufficient dilute nitric acid to dissolve the salt, and then evaporated each lot to dryness on the water-bath. When dry the two cells, and a third containing some of the original nitrate, were put on a sensitive plate for twenty-four hours.

On development it was found that the action of the part undissolved by ether was very strong, that of the original nitrate not more than half as strong, while no action whatever could be detected on the part of the plate covered by the salt soluble in ether.

The portion insoluble in ether, after evaporation to dryness with nitric acid, and then crystallisation in water, in no way differed in appearance from ordinary uranium nitrate. The portion soluble in ether, when dried, heated with dilute nitric acid, and crystallised, also had the same appearance as the initial salt.

14. The crystallised nitrate from the portion insoluble in ether I again extracted with ether. Most of it dissolved, and a small portion of heavy aqueous liquid settled at the bottom. As before, the nitrate which dissolved in ether had scarcely any radio-active power, while the residue from this second extraction possessed it in a strong degree. The residue after the second extraction was about double the activity of the residue after the first extraction, showing that ether, while dissolving uranium nitrate itself with facility, does not dissolve the body to which it owes its radio-activity.

15. The uranium nitrate from the portion insoluble in ether was submitted to fractional crystallisation in the following manner:—

The solution was evaporated until, on cooling, about three-fourths would crystallise out. The beaker in which this operation was performed was called No. 1. When crystallisation had finished, the mother-liquor was poured into a beaker called No. 2. A little water was added to No. 1 and it was warmed to dissolve the crystals; No. 2 was evaporated a little, and both were set aside to crystallise. When cold the mother-liquor from 2 was poured into a beaker No. 3, the mother-liquor from 1 was poured into 2, a little water being added to dissolve the crystals in 1, and the contents of the three beakers were warmed and allowed again to crystallise separately. This operation was continued as long as the uranium salt would hold out, or till the tests showed that the operations had gone far enough.

16. Tests were made to see how the operations were proceeding. A portion of the crystals from No. 1 beaker was dried and put into a cell. Some mother liquor from the last beaker was also evaporated and crystallised, and the crystals were put in a cell. The two were placed side by side on a sensitive plate and the action was allowed to proceed for twenty-four hours. On development there was no visible spot beneath No. 1 nitrate, while that beneath the nitrate from the other end of the fractionation was strong and black.

17. The crystals were removed from their cells, ignited to the green oxide, and replaced. Tested again on a sensitive plate, the results were similar to those given by the unignited nitrates. The active substance, therefore, is seen to reside in the mother-liquor.

(To be continued).

Reaction between Strychnine and Iodide of Methylene.—P. F. Trowbridge.—When heated for an hour in a sealed tube in the presence of methylic alcohol, or shaken up for several days with a chloroformic solution of the alkaloid, strychnine and iodide of methylene react one on the other, giving a white crystalline compound having the formula $C_{21}H_{22}N_2O_2.CH_2I_2$, melting at 212°. By digesting its aqueous solution with freshly precipitated chloride of silver we obtain the corresponding chlorised derivative in the form of white needles, very soluble in water, with which double salts of gold, mercury, and platinum can be prepared. The bromide can be obtained in a similar way by means of the chloride, by agitating the warm aqueous solution of the latter with oxide of silver, and slightly acidulating the filtrate with hydrobromic acid. This body appears in the form of white needles.—*Archiv der Pharmacie*, ccxxxvii., p. 617.

ON THE PROGRESS OF CHEMISTRY IN GREAT BRITAIN AND IRELAND DURING THE NINETEENTH CENTURY.*

Part I.—From the Beginning of the Century to the Foundation of the Chemical Society (1841).

By T. E. THORPE, LL.D., F.R.S.,
President of the Chemical Society.

(Concluded from p. 249).

In the year of Davy's death, Edward Turner wrote "The Æra of Brilliant Discovery in Chemistry appears to have terminated for the present." Although the number of workers in the science was probably greater than during the first few years of the century, the volume of chemical literature actually produced, judged by the number of contributions to the various societies and to scientific periodicals, had been steadily declining during the previous ten years, and was actually not much more than half what it was in 1802. The work of the first quarter of the century had, in fact, been that of the pioneers, and it was now necessary to secure and consolidate what had been gained rather than to push on into new territory. "The time is arrived," continues Turner, "for reviewing our stock of information, and submitting the principal facts and fundamental doctrines of the science to the severest scrutiny. The activity of chemists should now, I conceive, be especially employed, not so much in searching for new compounds or new elements, as in examining those already discovered; in ascertaining with the greatest possible care the exact ratio in which the elements of compounds are united; in correcting the erroneous statements to which inaccurate observation has given rise; and exposing the fallacy of opinions which partial experience or false facts have produced." Stoichiometrical considerations were, in fact, becoming of increasing importance. The atomic theory was now a part of the settled doctrine of chemistry. To use the words of Davy, chemistry had now passed under the dominion of the mathematical sciences. The exact numerical relations of the elements and their combinations, both by weight and by volume, Prout's hypothesis and all that it implied, were types of the questions which seemed most urgently to require solution. The idea of stating the course of chemical change with a more mathematical precision than had hitherto been possible is reflected, too, in the attempts which were made at about this period to express such changes by means of equations. Chemical symbols have been used from time immemorial, but the notion of attaching a definite numerical value to them had a rational basis only after the promulgation of the atomic theory.

Berzelius is usually credited with having been the first to suggest, in principle, the system of notation at present employed by chemists, although there is a tradition in Glasgow that the merit should be ascribed to Dr. Thomas Thomson. Herschel, in his paper on "Hyposulphurous Acid," in the *Edinburgh Philosophical Journal* for 1819, uses algebraic expressions to describe the reaction between silver nitrate and calcium thiosulphate. The idea of using a mathematical notation to express the chemical composition of substances and their mutual actions, although in common use in Sweden, France, and Germany, for a time made little progress in England, partly from the conservatism of chemists, who, like Richard Phillips, failed to perceive its necessity or convenience, and to whom the language of symbols—

> ". . . . was a Babylonish dialect
> Which learned *chemists* much affect:
> It is a party-coloured dress
> Of patch'd and piebald languages;
> 'Tis English cut on Greek and Latin,
> Like fustian heretofore on satin;

and partly from the strictures of mathematicians, chief

* Presidential Address to the Chemical Society, 1900.

among whom was Whewell, who condemned the Berzelian system for what he regarded as its "gross anomalies" and "disfigurements," and its want of "mathematical symmetry and consistency"—"a system which violates mathematical propriety so entirely that it must always be disagreeable to see an example of it, for any person who has acquired the first rudiments of algebra."

Whewell, whose "forte was science, and whose foible was omniscience," of course recognised that a mathematical notation might be of the greatest service in chemistry, and, indeed, could not ultimately be dispensed with, and he made a vigorous attempt "to purify and improve the foreign system." One of the chief rocks of offence to Whewell was the method adopted by Berzelius of connecting the symbols of the elements to represent compound substances. Chemical combination is essentially an operation of addition, whereas the symbolic notation, if understood algebraically, would indicate that the constituents were multiplied by each other. There is not much force in this objection, although, as Whewell points out, we probably owe to the symbols being read algebraically the ambiguity of the words *factor* and *product*, which are sometimes used to express the ingredients producing a chemical compound by their addition, as well as the compound itself, but which in algebra properly refer to parts producing a number by multiplication. Still more objectionable to Whewell was the use of the sign +; this he characterised as "a wanton and superfluous violation of analogy."

Whewell's objections were rather more pedantic than practical. It was pointed out that the juxtaposition of symbols and the employment of an index figure, as practised by Berzelius, would lead the chemist into no error, because their subjects are not susceptible of algebraic *powers*, or of being multiplied into each other.

On general grounds, too, Whewell's system failed to commend itself to chemists, even to those who were not unmindful of the mathematical proprieties. It only approximated to mathematical consistency, and failed altogether as regards brevity and clearness. The multiplication of lines and brackets which it entailed, although algebraically just, gave a perplexing appearance to the formula, and destroyed its graphic character. The unvarying sign + alternately with every letter was also objectionable. Whewell, who was not insensible to these defects in his system, suggested a method of contraction, but the compromise failed to realise the graphic perspicuity of the Berzelian notation, whilst, as an analytical expression, it was inferior to the extended system from which it was evolved. Whewell, moreover, avowedly put it forward as more suitable for the purposes of mineralogy than of chemistry, and the objection was naturally raised that a system which required new contrivances and contractions to suit different points of reasoning, and was so partial and so incomplete an expression of chemical composition in general, was certain to lead to interminable confusion and difficulty.

The serial chemical literature of the early thirties contains a number of interesting papers on chemical notation, not only by Whewell, but by Phillips, Prideaux, Warington, and Graham, and the merits of the various systems proposed were fairly discussed, with the result that the Berzelian method was at length universally adopted. As an example of the confusion which reigned at the time, we give, from one of Phillips's papers, the composition of common phosphate of soda as expressed in the different systems of notation then in vogue.

With the exception of Graham, it cannot be said that British or Irish chemists made any very remarkable contributions to science during the decade from 1830 to 1840. The number of workers was doubtless greater than at any previous period of the century. The volume of literature, as measured by the number of papers published, was more than double that of the first decade—one of the most momentous periods of our chemical history. But the efforts of workers at the time were spent more on

Berzelius	$\dot{N}a\dot{P}+24$ Aq.
,, ...	$\dot{N}a^2\dot{P}+24\dot{H}$.
Graham	$\dot{N}a^2\dot{H}^{24}\dot{P}$.
Rose	$NaO+PO^5+24HO$.
Whewell	$N+\dot{p}'+24$ q.
Brande	$S+\dot{p}'+24$ q.
Turner	$\dot{S}o+P+24O+24$ aq.
Johnstone	$\dot{P}+\dot{S}O+24\dot{H}$.
Prideaux	$\dot{N}\dot{P}+24$ *Aq*.
Warington	$\overset{\infty}{P}o+\overset{\circ}{S}o+24H^{\circ}$.

points of detail, on the filling in of little gaps in the chemical structure, as it then existed, than in attempts at new developments.

Thomas Clark, mainly remembered for his method of determining the hardness of water, and for his discovery of pyrophosphoric acid, was studying the metallurgy of iron. Apjohn, whose name is best known in connection with his formula for determining the dew-point from the indications of the wet bulb hygrometer, contributed a few papers on mineral chemistry; Daubeny, a professor of chemistry to botanists, and a professor of botany to chemists, occupied himself principally with geological and botanical chemistry; Colonel Yorke studied the action of air and water on lead; Everitt investigated the behaviour of dilute sulphuric acid with potassium ferrocyanide, and discovered the salt which is associated with his name; Johnston made analyses of mineral substances of organic origin, investigated paracyanogen, certain cyanides, and double haloid salts; Kane analysed a few minerals and inorganic salts, the chlorides of iodine, and studied the action of ammonia on the chlorides and oxides of mercury, the composition of essential oils, and the nature of wood spirit. Penny made a remarkably accurate series of atomic weight determinations of chlorine, nitrogen, potassium, and sodium, which served still further to strengthen the case against the hypothesis that all equivalent numbers are simple multiples of that of hydrogen; and Gregory, the successor of Graham at the Andersonian College, a follower of Liebig, and one of the earliest chemists to introduce his methods into Britain, occupied himself with the study of pharmacological and organic products, the analysis of petroleum, and the destructive distillation of caoutchouc. Other chemical workers at this period were Pepys, Porrett, Edmund Davy, Brooke, Cooper, Daniel, John Mercer, William Herapath, Walter Crum, William West, Hennell, Scanlan, Teschemacher, Hugh L. Pattinson, Warington, Andrews, Richardson, Robert D. Thomson, Denham Smith, Golding Bird, Solly, Fownes, Stenhouse, George Wilson, and Griffin.

But the general condition of scientific chemistry with us during this period was, no doubt, accurately characterised by Liebig, when he wrote to Berzelius in 1837:—"Ich bin einige Monate in England gewesen, habe ungeheuer viel gesehen und wenig gelernt. England ist nicht das Land der Wissenschaft, es existirt dorten nur ein weitgetriebener Dilettantismus, die Chemiker schämen sich Chemiker zu heissen, weil die Apotheker, welche verachtet sind, diesen Namen an sich gezogen haben.... Graham macht auch in wissenschaftlicher Hinsicht die schätzbarste Ausnahme, er ist ein vortrefflicher Mensch, auch Gregory, der an seine Stelle in Glasgow gekommen ist."

It was at this low ebb in our fortunes that the Chemical Society was founded. It might seem, at first sight, an unpropitious time at which to launch a new Society for the purpose of publishing the work of chemical investi-

gators, when chemical investigation itself was so languidly pursued. But the seventy-seven gentlemen from all parts of the kingdom who enrolled themselves as the founders of the Chemical Society appeared to have had a robust faith in its ultimate success, and in the influence it was bound to exercise in stimulating chemical inquiry in these islands.

How far their faith was well founded—how far indeed this Society has succeeded in stimulating chemical research in this kingdom—I may possibly have an opportunity of showing on some future occasion. I hope also to be able to offer some account of the development of our means of teaching chemistry in this country during the century.

ON

BLACK PHOSPHORUS, AND THE CONVERSION OF PHOSPHORUS INTO ARSENIC.*

By T. FITTICA.

In a paper on "Black Phosphorus" F. A. Flückiger expressed his conviction that this so-called modification consists of nothing but arsenic, and he explains its occurrence in ordinary phosphorus, respectively, its apparent formation from phosphorus by means of ammonia, on the assumption that ordinary phosphorus contains an admixture of arsenic, the phosphorus dissolving in ammonia with the production of PH_3, the arsenic remaining intact. My investigations, however, lead me to the conclusion that, by acting upon phosphorus with ammonia in the presence of air, a true transformation of phosphorus into arsenic takes place, and that the latter appears to be a nitrogen-oxygen compound of phosphorus.

In the first place, I confirmed the observation that, on treating ordinary phosphorus with concentrated (20 per cent) ammonia on the water-bath at about 60° C. (the phosphorus therefore in the fused state), with frequent shaking with free access of air, it is transformed into the black modification; that is to say, converted into arsenic. The operation is best carried out in a flask with a flat bottom and of considerably larger capacity than is necessary for the quantities of substances employed, in order that a large surface be presented to the air. The flask at the same time should carry a long glass tube, to prevent, as much as possible, the evaporation of the ammonia. The flask is from time to time removed from the water-bath for a short time and strongly shaken, in order to bring the fluid into contact with the air. The amounts of arsenic thus obtained are, however, but very small, and I therefore employed stronger oxidants than the oxygen of the air, taking hydrogen peroxide in the first instance.

When I warmed phosphorus with concentrated ammonia, and then added a recently prepared solution of hydrogen peroxide, allowing the mixture to stand at ordinary temperature for some length of time, I obtained somewhat larger quantities of arsenic. Even here, however, the amounts of arsenic produced were still very scant, and not sufficient for the confirmation of my suspicion that I had not to do with the detection of a contamination of phosphorus with arsenic, but with the real formation of the latter.

In order to obtain a secure basis for my conclusions I first resolved to examine the phosphorus, both ordinary colourless and amorphous red phosphorus, for its contents in arsenic. I found, however, that from equal amounts of phosphorus I obtained various quantities of arsenic, whether I used colourless or red phosphorus, or whether I used various oxidants. Generally the red phosphorus gave a larger proportion of arsenic by the usual oxidation with nitric acid, and especially when employing barium peroxide with hydrochloric acid or nitric acid, or potassium chlorate and hydrochloric acid. The same sample of colourless phosphorus which with dilute nitric acid gave a hardly noticeable trace of arsenic, yielded with concentrated nitric acid very plainly discernible quantities of arsenic, and larger ones still with barium peroxide and nitric acid. (0·4274 grm. phosphorus yielded 0·0142 grm. arsenious sulphide, equal to 2·02 per cent As; 0·501 grm. phosphorus yielded 0·2012 grm. arsenic sulphide, equal to 2·5 per cent arsenic). [*Note by Translator.*—There is evidently an error in these figures]. From amorphous phosphorus upwards of 2·6 per cent arsenic can thus be obtained (1·3981 grms. yield 0·049 grm. As_2S_3, equal to 2·13 per cent As; 0·2266 grm. gave 0·010 grm. As_2S_3, equal to 2·64 per cent As).

Oxidation with barium peroxide and hydrochloric acid gave smaller yields, but I did not succeed in obtaining agreeing results on employing various quantities of the same substances. But when finally 1·02 grms. of the amorphous phosphorus was oxidised by carefully mixing it with barium peroxide, and heating the mixture with dilute sulphuric acid, or on adding to the mixture water and concentrated sulphuric acid (to 1 grm. P 13·63 grms. BaO_2 and 8 grms. H_2SO_4) a very small trace of precipitate obtained by sulphuretted hydrogen from the product of complete oxidation from which the barium sulphate had been filtered off, proved to be pure sulphur, completely soluble in CS_2. The precipitate extracted with ammonia yielded a trace of substance which, however, was also completely soluble in CS_2, and burned upon a platinum foil with a blue flame.

It was obvious therefore that the production of arsenic from the phosphorus employed was due to the nitric oxidants, and to the employment of ammonia, and I therefore tried to effect a synthesis of arsenic by using both nitric acid and ammonia together, and also by employing nitrites. If ordinary phosphorus is heated on the water-bath in a tube connected with a condenser, with ammonium nitrate, no reaction takes place below 60°, but the action then becomes so violent that explosions may take place, even in presence of ammonium carbonate, corresponding with the well-known fact that phosphorus burns in ammonium nitrate vapour, just as it does in a current of oxygen. When phosphorus is heated together with ammonium nitrate and potassium nitrite in presence of ammonium carbonate, an exceedingly violent reaction takes place at a temperature approaching 100°. *Amorphous* phosphorus, however, appeared more suitable for the synthesis, and after many fruitless experiments I ultimately arrived at the following process, which as far as I can at present say, gives a yield of from 8 to 10 percent of the crude product:—

2 grms. amorphous phosphorus, free from arsenic as tested above, are carefully mixed with 12·9 grms of finely powdered ammonium nitrate, and the mixture is placed in a tube which must not be too narrow, and is heated on a sand-bath, the temperature slowing rising to 180°. A reaction ensues which must be moderated, if necessary, by removing the flame from the bath. The temperature is then gradually increased to 200°; when the reaction is finished, the mass is allowed to cool, dissolved in water, the solution filtered and precipitated with sulphuretted hydrogen. The precipitate is dissolved in ammonium carbonate, and the arsenic sulphide precipitated from the solution can be identified by its behaviour with ammonia and hydrochloric acid, by its oxidation into arsenic acid, and precipitation with a magnesia solution, as well as by testing of the magnesia precipitate in a Marsh apparatus. In accordance with the decomposition of ammonium nitrate on heating, and with the above stated proportions of phosphorus and ammonium nitrate, the formation of arsenic from phosphorus appears to take place mainly in accordance with the following equation:—

$$2P+5NH_4NO_3 = (PN_2O)_2O_3+10H_2O+3N_2.$$

But I have to remark, that besides this main reaction at least *one* secondary reaction takes place, as shown by the

* *Leopoldina, Halle*, vol. xxxvi., No. 3, March, 1900.

production of white fumes of neutral reaction. These fumes can be collected in water in which they dissolve. They contain phosphorus, and with sulphuretted hydrogen yield free sulphur. In accordance with the above equation arsenic would be a nitrogen-oxygen compound of phosphorus, corresponding to the formula PN_2O.

THE COMMERCIAL ANALYSIS OF COPPER.

By A. HOLLARD.

The copper is separated and estimated by the electrolytic method. The impurities are separated in the following manners:—The antimony, nickel, cobalt, silver, and lead electrolytically; the arsenic by distillation; the sulphur and gold by precipitation; the iron is estimated volumetrically.

The apparatus used has already been described and illustrated (*Bull. Soc. Chim.*, Series 3, xxxiii., p. 291). According to the nature of the element to be deposited, the surface of the electrode must be polished or rough. Copper and lead (in the state of oxide) deposit best on a rough surface; antimony, nickel, and cobalt on a smooth surface; and silver on either.

Details of the Electrolysis.

Estimation of the Copper (*Electrolysis in Acid Solution*).—10 grms. of the granulated material are taken and freed from grease and particles of iron by means of ether and a magnet. The material is then placed in a 500 c.c. beaker, 6·5 c.m. diameter, and 18 c.m. high. To this are added 20 c.c. of sulphuric acid, and 30 c.c. of nitric acid, sufficiently diluted with water to prevent a too violent reaction. It is then covered with a funnel, stem downwards, and heated gently. With fine copper the solution is complete, but unrefined metal leaves an insoluble residue; in such a case the solution must be further heated to collect the precipitate, and to free it from copper salts. The solution is diluted to 200 c.c., and the electrodes, connected to the two poles of a battery, completely submerged in it. The beaker is covered with two half clock-glasses, and a current of 1 ampère passed through.

When the solution contains no more copper the beaker and the solution are removed, and the electrodes plunged rapidly into two glasses of distilled water; the one covered with copper is next plunged into strong alcohol, then still wet with alcohol, placed in an oven, and dried at 90° for about ten minutes, and weighed. The weight found, minus that of the electrode, is the weight of the copper, plus the silver deposited at the same time. The weight of the copper can thus be deduced when we know the weight of the silver which is determined later on. If the copper contains lead, only part of this latter will be deposited as oxide, the remainder keeping in solution. The precipitation of the copper under these conditions takes about twenty-four hours.

We must observe that the complete electrolysis of copper under the given conditions can only be effected with coppers very poor in arsenic and antimony. As a general rule the quantities of arsenic and antimony present are sufficiently high for them to be partially precipitated with the last traces of copper. In such cases the electrolysis is stopped when the blue colour begins to disappear, that is when less than 50 m.grms. of copper remain in the bath, and the copper which also contains the whole of the silver is weighed. The remainder of the copper is estimated, as described further on, after the separation of the arsenic and antimony, but whether all the copper has been removed or not, the analysis is continued as follows:—

Estimation of the Antimony (*by Electrolysis in Cyanide Solution*).—15 c.c. of hydrochloric acid is added to the solution, which is then evaporated to dryness. On cooling the residue is taken up with dilute hydrochloric acid, heating to complete the solution. The solution, diluted to 100 c.c., is then saturated in the cold, with hydrochloric acid gas, and then heated with 200 c.c. of pure ordinary hydrochloric acid saturated with sulphuretted hydrogen. This precipitates nearly the whole of the arsenic, the copper and antimony remain in solution. After twelve hours it is filtered with a filter-pump through glass-wool covered with asbestos. The sulphide of arsenic thus separated is not kept, the estimation of the arsenic being made on another sample of the copper. 300 c.c. of pure water, and 300 c.c. of water saturated with sulphuretted hydrogen, are then added to the solution. The copper and antimony are thus completely precipitated. After standing for a few hours the sulphides of these two metals are filtered, washed with a solution of sulphydric acid, then dissolved in a mixture of cyanide of potassium at 20 per cent (62·5 c.c.), and concentrated sulphide of sodium (20 c.c.) (density 1·22, and prepared according to Classen). The solution thus obtained is diluted to 220 c.c. in a beaker 6 c.m. in diameter; the electrodes are completely immersed therein, and a current of 0·05 ampère passed for a whole night. The antimony is deposited integrally, and forms a very adherent steel-grey metallic layer. To obtain an adherent deposit the amount of antimony to be precipitated must not surpass 0·05 grm.

Precipitation of the Remainder of the Copper.—The copper remaining, with a few milligrms. of arsenic, after the separation of the antimony, is precipitated electrolytically in the following manner:—Sulphuric acid is added until a precipitate is formed; it is then heated to drive off the sulphuretted hydrogen, and bromised hydrochloric acid added; this dissolves the precipitate. Evaporate to dryness, and take up with 10 c.c. of nitric acid and water; when the solution is effected, add 10 grms. of nitrate of ammonia, and dilute to 100 c.c. This solution is then electrolysed with a current of 1 ampère. For very small quantities of copper the colorimetric method may be used.

Estimation of Nickel and Cobalt (*by Electrolysis in Solution of Double Ammoniacal Sulphate*).—The liquid freed from copper by electrolysis, and from arsenic and antimony by sulphuretted hydrogen is heated to drive off the H_2S; the iron is then peroxidised by adding nitric acid, and boiling the solution; the latter is then evaporated to dryness on a sand-bath until white sulphuric fumes appear. The residue is taken up with dilute sulphuric acid, and then with water to dissolve everything. It is then treated with ammonia, and more sulphuric acid if necessary, so that each 100 c.c. of solution will contain 8 to 11 c.c. of ammonia combined with sulphuric acid. A slight excess of ammonia is then added to precipitate the iron; after boiling, the solution is allowed to cool, and 12 to 20 c.c. more of ammonia at 10 per cent is added per 100 c.c. of solution. Dilute to 250 c.c., and allow the peroxide of iron to collect at the bottom of the beaker. The electrodes are completely immersed in the clear liquid, and a current of 0·5 ampère passed. After a night the nickel and cobalt are completely precipitated. Wash and dry the electrode A as described for copper, then weigh. If the proportions of nickel and cobalt are wanted separately, they can be estimated by any known method. The maximum quantity of metal to have an adherent deposit is 0·2 grm.

Volumetric Estimation of the Iron.—The peroxide of iron is thrown on a filter, then dissolved in the smallest possible quantity of hydrochloric acid, and estimated by Mohr's iodine method.

Estimation of the Lead (*by Electrolysis in Acid Solution*).—A fresh sample of 10 grms. of copper is attacked by dilute nitric acid containing 50 c.c. of acid at 36° B. The liquid, filtered if necessary, and diluted to 350 c.c., is submitted to electrolysis; the intensity of the current should be 0·3 ampère. After eighteen hours the lead is precipitated integrally on the positive electrode A in the state of oxide in a very adherent deposit, brown or black according to the thickness, while the copper is partially deposited on the negative electrode B. The electrode A is then

plunged successively into two vessels filled with distilled water, then placed in an oven which is gradually heated to 200°. Under these conditions the oxide of lead corresponds exactly to the formula PbO_2; it is sufficient to multiply its weight by 0·866 to get the corresponding weight of metallic lead.

Estimation of the Silver (by Electrolysis in a Cyanide Solution, and Volumetrically).—If the copper is rich in silver, the copper deposited electrolytically on the electrode A is dissolved in nitric acid of 1·2 density; we know it must contain all the silver. In the opposite case we dissolve a fresh sample of 10 to 50 grms. of copper, according to the supposed amount of silver present; boil to drive off the nitrous fumes, filter if necessary, and precipitate the solution at 70° with a few drops of hydrochloric acid; this temperature is maintained until the precipitate collects. Filter, wash with warm water, and re-dissolve the precipitate in cyanide of potassium, and dilute to 250 c.c.; the solution should contain 2 per cent of cyanide of potassium. The current should be 0·05 ampère. After one night the precipitation is complete. This precipitate might be weighed, but it is better to dissolve it in nitric acid diluted with its own volume of water, and titrate by Volhardt's sulphocyanide method. But in order that the precision of this method may be greater than that by weighing, the end of the reaction must be determined, not as Volhardt says, by the appearance of the red colouration due to ferric sulphocyanide, but by titrating back by means of titrated nitrate of silver from this red colouration to the white appearance of sulphocyanide of silver. The distinctness of this latter reaction is incomparably greater.

The silver is therefore dissolved in about 100 c.c. of a mixture of equal volumes of nitric acid and water, and heated to boiling to drive off all nitrous fumes. The cooled solution is treated with 5 c.c of ammoniacal iron alum at 20 per cent then with a titrated solution of sulphocyanide of ammonium until the red colour appears. A titrated solution of nitrate of silver (2 grms. of silver per litre) is then added until the red colour passes to pink, and then becomes *suddenly* white. The titrated solutions of sulphocyanide of ammonium and of nitrate of silver must correspond exactly, so that it will suffice to subtract from the number of c.c. of sulphocyanide used the number of c.c. of nitrate of silver added, and multiply this difference by 2 to obtain the weight of the silver in milligrms.

Estimation of the Arsenic (by Distillation and Volumetrically).—In a flask of 300 c.c. capacity we place 5 grms. of the granulated metal with 50 grms. of ferric sulphate.* Then, through a funnel provided with a stop-cock, we add 150 c.c. of pure hydrochloric acid; the flask is connected with a U-tube immersed in an oil-bath, and thence through a bent tube the gases pass into a test-jar. After pouring the acid into the flask the tap is closed, and after seeing that the oil-bath is at 150° to 175° the flask is gently heated. The metal dissolves, and the arsenic distils as arsenious chloride; this chloride is collected in the test-jar containing 50 c.c. of water. The operation is stopped in about half an hour, when 35 c.c. of the chloride has passed over. Under these conditions the arsenic alone passes into the test-jar, the antimony remaining in the flask, and in the U-tube which contains glass beads: this tube further retains all the projections which might be carried over from the flask. The arsenious solution obtained by distillation is titrated by iodine as follows:—Ammonia is added to the cooled solution until alkaline, it is then re-acidulated by a few drops of hydrochloric acid, and a slight excess of bicarbonate of soda added. The solution is then cooled again, 5 c.c. of 1 per cent starch added, and titrated with iodine until the blue colour is persistent. The titrated solutions are prepared in the following manner:—

* This sulphate must be freed from all trace of nitrous fumes by evaporation in the presence of an excess of sulphuric acid, after having been finely powdered.

Arsenious Acid.—Weigh out 3·3 grms. of pure arsenious acid (= 2·5 grms. arsenic) in fine powder, and 9 grms. of bicarbonate of soda. Treat the mixture with 500 c.c. of boiling water, and prolong the boiling until all the arsenious acid is dissolved. Cool, add 2 grms. of bicarbonate of soda, and make up to 1 litre, 1 c.c. = 0·0025 grm. of arsenic.

Solution of Iodine.—Dissolve 3·3 grms. of pure iodine in 50 c.c. of a 20 per cent solution of iodide of potassium, dilute to 1 litre, and titrate with 20 c.c. of the arsenious acid solution; 1 c.c. of the solution of iodine should correspond with 0·001 grm. of arsenic. Care must be taken to make all the estimations on the same volume of liquid, and to observe the number of tenths of a c.c. necessary to obtain a permanent blue colour in a solution free from arsenic. The titration of the solution of iodine should be repeated occasionally.

Solution of Starch.—Boil 10 grms. of potato starch in a litre of water. Add 1 grm. of potash alum as an antiseptic, and after cooling, decant the clear liquid. This solution will keep for several months.

Estimation of the Gold (in the Metallic State).—100 grms. of copper are dissolved in 750 c.c. of nitric acid of 1·2 density, boil to drive off the nitrous fumes, and filter. The filter which contains all the gold is dried, and then burnt. The residue is cupelled with lead and a little silver. The silver button, obtained by cupellation, containing all the gold is dissolved in nitric acid of 1·2 density, the gold which remains undissolved is dried and weighed.

Details of the Operation.—The filter with its contents are placed on a small sheet of thin, pure, lead about 7 c.m. square, weighing about 20 grms. The paper is set fire to, and left to burn alone; after the combustion a few centigrms. of silver are placed on the lead, and the whole wrapped up tightly, and placed on a cupel in a red-hot muffle. After cupellation the flattened button is placed in a matras with nitric acid (free from chlorine), of 1·2 density; the slightly inclined matras is heated gently until all nitrous fumes are driven off. The clear liquid is then decanted with great care, and the matras partially filled with pure water and again decanted. If necessary the washing may be repeated; finally, the matras, filled with water, is inverted into a small clay crucible; when the gold has fallen into the crucible the matras is removed, the water in the crucible is poured off, and the crucible dried in front of the muffle; when dry it is placed in the muffle, and heated strongly for a few minutes. On cooling, the gold, which should be of a yellow colour, is placed on the balance and weighed.

Estimation of the Sulphur (in the State of Sulphate of Barium).—From 5 to 20 grms. of copper, according to its richness, are attacked with aqua regia, with an excess of nitric acid, and the sulphur in the solution is estimated by the known method.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 8.

Estimation of Acetic Acid in Commercial Acetates, and its Separation from Propionic, Butyric, and Formic Acids.—R. Haberland.—The four acids are set free by means of phosphoric acid, and distilled in a current of steam. The distillate is treated with PbO and evaporated; the residue is taken up with water, and the basic propionate of lead is left in the insoluble state. The filtrate is freed from lead by sulphuric acid, treated with ZnO, and evaporated to dryness. The residue is taken up with alcohol, which will not dissolve the acetate or butyrate. The alcoholic solution is evaporated to dryness, the mass treated with phosphoric acid, and the acids distilled; they are then transformed into silver salts, the salt of butyric acid being very slightly soluble in water.—*Zeit. Anal. Chem.*, xxxviii., p. 217.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 17th, 1900.

Professor THORPE, F.R.S., President, in the Chair.

MESSRS. John R. Brooke, J. Wallace Sandford, and Bertram D. Steele were formally admitted Fellows of the Society.

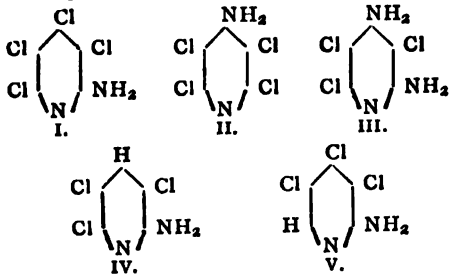
Certificates were read for the first time in favour of Messrs. Charles Berjew Brooke, Colne House, near Manningtree, Essex; Ernest Owen Courtman, Denford House, Atkin's Road, Clapham Park, S.W.; Henry Bertie Gritton, The Cottage, Balmain, Sydney, N.S.W., Australia; John Beresford Leathes, 10, Park Mansions, Battersea Park, S.W.; Sidney Scrivener Napper, 144, Highbury New Park, N.; Oswald Silberrad, Sunny Croft, Buckhurst Hill, Essex; John Traquair, Glenfield Starch Works, Paisley.

The following Certificates were approved by Council under Bye-law I. (3):—Angelo Cantin, Vacoas, Mauritius; M. C. Nanjunda Row, Mylapore, Madras; M. Goolab Roy, Chintadrepettah, Madras.

Of the following papers, those marked * were read:—

*67. "*The Chlorine Derivatives of Pyridine.* VI. *The Constitution of some Aminochloropyridines.*" By W. J. SELL, M.A., and F. W. DOOTSON, M.A.

The authors gave an account of their work on the constitution of various aminotrichloro- and aminotetrachloropyridines which have recently been described by them. The two compounds which result from the action of ammonia on pentachloropyridine (*Trans.*, 1898, lxxiii., 777), and which melt at 174—175° and at 212—213°, are shown to have the constitutions represented by the formulæ I. and II. respectively.



I. II. III.

IV. V.

Both compounds heated with ammonia to a higher temperature give the same diaminotrichloropyridine, which must therefore be represented by formula III.

To the aminotrichlorpyridine, obtained by the action of ammonia on tetrachloroisonicotinic acid (*Trans.*, 1897, lxxi., 1083), the constitution represented by formula IV. was provisionally assigned (*Trans.*, 1899, lxxv., 980). This has now been completely established.

The aminotrichloropyridine, obtained (*Trans.*, 1899, lxxv., 980) by the action of sodium carbonate on a compound containing two pyridine rings, was shown to have the constitution represented by formula V.

*68. "*Ortho-substituted Nitrogen Chlorides and Bromides, and the Entrance of Halogen into the Ortho-position in the Transformation of Nitrogen Chlorides.*" By F. D. CHATTAWAY and K. J. P. ORTON.

When phenylacetyl nitrogen chloride undergoes transformation, a mixture of *o*- and *p*-chloroacetanilide is produced in the proportion of 95—96 per cent of the latter together with 4—5 per cent of the former.

The following compounds were described:—

Orthochlorophenylacetylnitrogen chloride (acetyl chloramino-2-chlorobenzene), $C_6H_4Cl{\cdot}NCl{\cdot}COCH_3$, colourless, long, flattened prisms, m. p. 88°.

Orthobromophenylacetylnitrogen bromide (acetyl bromamino-2-bromobenzene), $C_6H_4Br{\cdot}NBr{\cdot}COCH_3$, yellow, four-sided prisms, m. p. 150—152°.

Both of these compounds undergo transformation into the 2 : 4-disubstituted acetanilides.

DISCUSSION.

Dr. HEWITT pointed out that Beilstein and Kurbitow (*Annalen*, 1875, clxxvi., 36) had separated the ortho- from the para-chloraniline by distilling the mixed sulphates with steam, this method being employed by them on account of the difficulty of isolating pure *o*-chloronitrobenzene from the mixture obtained on nitrating chlorobenzene.

Dr. COLLIE pointed out that if a compound were obtained in which the halogen was attached to a nitrogen atom which was not directly united to the benzene nucleus, it was possible that isomeric change involving the production of a meta-substitution product might result.

*69 "*Ammonium Imidosulphite.*" By EDWARD DIVERS and MASATAKA OGAWA.

The authors have discovered a compound, $3NH_3.2SO_2$, which has the constitution $HN(SO_2NH_4)_2$, and bears the same relation to the amidosulphite as that which exists between imidosulphate and amidosulphate. When ammonium amidosulphite is allowed to decompose completely in a current of well dried hydrogen or nitrogen at a temperature not exceeding 35°, it leaves a coloured residue which is practically insoluble in absolute alcohol. This residue, freely soluble in water, can (with the exception of a small quantity of sulphate) be dissolved by repeated treatments with alcohol of 90 per cent. Rejecting the coloured fractions first obtained, the remainder of the solution on evaporation over sulphuric acid deposits bright micaceous crystals of the new salt *ammonium imidosulphite*. The mother liquor, which contains much ammonium amidosulphate, must be removed by careful washing with 95 per cent alcohol. The imidosulphite is very deliquescent, and all samples so far prepared have an acid reaction towards litmus, due to the loss of a little ammonia. By dissolving the ammonium salt in 75 per cent alcohol and adding exactly the equivalent quantity of alcoholic potash, the pure crystalline *potassium imidosulphite*, slightly alkaline to litmus, is obtained. The imidosulphites react with silver nitrate similarly to the amidosulphites, and give with barium chloride a precipitate which readily dissolves in acids, forming a solution which acquires the odour of sulphur dioxide, and deposits barium sulphate.

*70. "*The Constitution of Ethyl Sodiocyanacetate and of Ethyl Methylsodiocyanacetate.*" By JOCELYN FIELD THORPE.

The author pointed out that the reactions of ethyl sodiocyanacetate and of ethyl methylsodiocyanacetate are best represented by the formulæ—

$$CN{\cdot}CH{:}C(ONa)OEt \text{ and } CN{\cdot}C(CH_3){:}C{<}^{ONa}_{OEt}$$

respectively.

When *ethyl sodiocyanacetate* is dissolved in water, the solution is not alkaline, and on evaporation in a vacuum leaves sodium cyanacetate. The changes involved may be represented thus:—

$$CN{\cdot}CH{:}C{<}^{ONa}_{OEt} + H_2O =$$

$$= CN{\cdot}CH_2{\cdot}C{<}^{ONa}_{OH}_{OEt} = CN{\cdot}CH_2{\cdot}CO_2Na + HOEt.$$

The production of acid ethyl salts frequently observed in the condensation of ethyl cyanacetate with unsaturated ethyl salts can be thus explained, as the sodium compound of the condensation product, on treatment with water, would yield in a similar way the corresponding sodium ethyl salt. The acid ethyl salts on distillation lose carbon dioxide, and pass into the corresponding nitriles.

A number of such condensations were described, and the conditions given under which the maximum quantity of acid ethyl salt is produced. Experiments were described showing that prolonging the time of heating increases the quantity of neutral ethyl salt at the expense of the acid ethyl salt. This is explained by assuming that the first step is the addition of a molecule of alcohol to the unsaturated sodium compound.

Ethyl methylsodiocyanacetate behaves in the same way as ethyl sodiocyanacetate, but its solubility and deliquescent nature render its purification difficult. It condenses with unsaturated ethyl salts to form acid ethyl salts, the yields of which, however, were never more than 17 per cent of those theoretically possible.

The equation $CN\cdot CHNa\cdot CO_2Et + CH_2{:}CCH_3\cdot CO_2Et = CN\cdot CH(CO_2Et)\cdot CH_2\cdot CNa(CH_3)CO_2Et$, usually supposed to represent a reaction of this kind, is shown to be wrong, because on treating the product of the reaction with methyl iodide, the ethyl salt obtained gives on hydrolysis the symmetrical *aa'*-dimethylglutaric acids, and not the unsymmetrical *aa*-dimethylglutaric acid.

Small quantities of *cis*- and *trans*-acids such as are obtained from ethyl cyanacetate and ethyl methylcyanacetate, may be separated by the following method:—The cyanoethyl salt is converted by treatment with methyl alcoholic potash into the cyano-acid, which is then boiled with concentrated hydrochloric acid as long as carbon dioxide is evolved. The *cis*- and *trans*-amides, the intermediate products formed, are finally converted, the former into the imide, the latter into the *trans*-acid. The imide is insoluble in dilute sodium carbonate solution, and can thus be separated from the *trans*-acid.

*71. "*The aa$_1$ββ-Tetramethylglutaric Acids.*" By Jocelyn Field Thorpe and W. J. Young.

Ethyl Sodiocyanacetate, $CN\cdot CH{:}C(ONa)OEt$, separates from absolute alcohol in lustrous plates; with water it gives (see preceding abstract) sodium cyanacetate; with an ice cold solution of iodine in ether, it reacts to form *ethyl iodocyanacetate*, $CN\cdot CHI\cdot CO_2Et$, a brown oil which decomposes on distillation under diminished pressure; an intermediate addition compound is, however, formed, which reacts with unchanged sodiocyanacetate at higher temperatures to form *ethyl dicyanosuccinate*, thus:—

$$CN\cdot CH{:}C\begin{matrix}ONa\\OEt\end{matrix} + CN\cdot CHI\cdot C\begin{matrix}ONa\\I\\OEt\end{matrix} = \begin{matrix}CN\cdot CH\cdot CO_2Et\\|\\|\\CN\cdot CH\cdot CO_2Et\end{matrix} + 2NaI.$$

Ethyl dicyanosuccinate separates from dilute alcohol in glistening plates which melt at 120°.

With bromine dissolved in carbon bisulphide, only *ethyl bromocyanacetate*, $CN\cdot CHBr\cdot CO_2Et$, was obtained as a brownish oil which decomposes on distillation under diminished pressure.

Ethyl methylsodiocyanacetate, $CN\cdot C(CH_3){:}C\begin{matrix}ONa\\OEt\end{matrix}$, is not precipitated on the addition of alcoholic sodium ethoxide to ethyl methylcyanacetate, but is obtained as a gelatinous precipitate on adding an equal volume of dry ether to the solution. It combines with iodine to form *ethyl methyliodocyanacetate*, $CN\cdot C(CH_3)I\cdot CO_2Et$, a brown oil decomposing on distillation under diminished pressure. It condenses with ethyl dimethylacrylate to give the acid salt, $CO_2H\cdot CH(CN)\cdot C(CH_3)_2\cdot CH(CH_3)\cdot CO_2Et$, which, on distillation, loses carbon dioxide and gives *ethyl α-methyl-ββ-dimethyl-γ-cyanobutyrate*,—

$$CN\cdot CH_2\cdot C(CH_3)_2\cdot CH(CH_3)\cdot CO_2Et,$$

a fairly mobile oil boiling at 224°, which, on hydrolysis, gives *aββ*-trimethylglutaric acid melting at 87°. The preparation of *ethyl α-cyano-aa$_1$ββ-tetramethyl glutarate*, b. p. 174—176°, and its conversion into *tetramethyl glutarimide* (m. p. 108°), and into trans-*aββa'-tetramethyl glutaric acid* (m. p. 98°) are described. The imide is converted by heating for two hours with 50 per cent sulphuric acid into the *cis*-acid which melts at 140°, and this, on treatment with acetyl chloride, yields its anhydride, a liquid boiling at 155—160° (30 m.m.). The *cis*-acid is partially converted into the *trans*-acid by heating it in a sealed tube with concentrated hydrochloric acid.

*72. "*β-Isopropylglutaric Acid, and the* cis- *and* trans-*Methyl Isopropyl Glutaric Acids.*" By F. H. Howles, J. F. Thorpe, and W. Udall.

The main facts regarding β-isopropylglutaric acid have already been described (*Proc.*, 1899, xv., 103).

By the action of methyl iodide on the sodium compound of the product resulting from the condensation of ethyl sodiocyanacetate with ethyl β-isopropylacrylate, *ethyl α-cyano α-methyl-β-isopropyl glutarate* is obtained as a thick oil boiling at 180—183° (25 m.m.). This on hydrolysis with methyl alcoholic potash yields the corresponding *potassium* salt, from the acidified aqueous solution of which the solid cyano-acid may be obtained; this, on boiling with concentrated hydrochloric acid, passes into a mixture of the imide and the *trans*-acid. *α-Methyl-β-isopropyl glutarimide* separates from water in colourless needles melting at 114—115°. Trans-*α-methyl-β-isopropyl glutaric acid* separates from water in microscopic crystals melting at 102°, and is converted into the anhydride of the *cis*-modification on heating with acetic anhydride in a sealed tube.

The *cis*-modification (obtained from the imide by heating with 50 per cent sulphuric acid) crystallises from water in glistening plates melting at 137°, and on treatment with acetic anhydride is converted into the anhydride which crystallises from light petroleum in prisms melting at 44°.

Ethyl sodiocyanacetate condenses with ethyl α-methyl acrylate to form the acid ethyl salt,—

$$CO_2H\cdot CH(CN)\cdot CH_2\cdot CH(CH_3)\cdot CO_2Et,$$

which on distillation yields *ethyl α-methyl-γ-cyanobutyrate*, a mobile oil boiling at 205°, giving on hydrolysis α-methylglutaric acid.

By a similar method to that above described *ethyl α-cyano-aa'-dimethylglutarate* (b. p. 181—183°), the *potassium salt* of the cyano-acid, the *cyano-acid* and the *cis*- and trans-*aa'-dimethylglutaric acids* were prepared.

Ethyl-β methyl-γ cyanobutyrate and *β-methylglutaric acid* have also been obtained in a similar manner.

*73. "*The Racemisation of Optically Active Tin Compounds. Dextromethylethylpropyl Tin Dextrobromocamphorsulphonate.*" By William Jackson Pope and Stanley John Peachey.

The authors have shown (*Proc.*, 1900, xvi., 42) that the aqueous solution obtained by treating externally compensated methylethyl-*n*-propyl tin iodide with aqueous silver dextrocamphorsulphonate yields only dextromethylethylpropyl tin dextrocamphorsulphonate on evaporation to dryness. They attributed this behaviour to progressive racemisation of the lævorotatory radicle, $(CH_3)(C_2H_5)(C_3H_7)Sn^{\cdot}$, attending crystallisation of the sparingly soluble salt of the dextrorotatory radicle. As this, the first well-determined case of the kind recorded, has an important bearing upon the space configuration of quadrivalent sulphur compounds (*Proc.*, 1900, xvi., 12), and upon the preparation of compounds owing their optical activity to the asymmetric nitrogen atom (*Trans.*, 1899, lxxv., 1127), it seemed to merit further investigation.

On treating externally compensated methylethyl-*n*-propyl tin iodide with silver dextro-α-bromocamphorsulphonate in warm aqueous solution and evaporating the filtered liquid, *dextromethylethylpropyl tin dextrobromocamphorsulphonate*,—

$$(CH_3)(C_2H_5)(C_3H_7)Sn(C_{10}H_{14}BrOSO_3),$$

separates in colourless plates which, after crystallisation from acetone, melt at 194—197° with previous softening; the mother-liquors yield, on evaporation, further deposits of the same salt, and no trace of salt of the lævorotatory base could be obtained. The behaviour of the dextro-

camphorsulphonate and the dextrobromocamphorsulphonate is thus the same, the whole of the lævorotatory base, $(CH_3)(C_2H_5)(C_3H_7)Sn{\cdot}OH$, becoming converted into its dextrorotatory isomeride. In dilute aqueous solution, the bromocamphorsulphonate has the molecular rotation +318°, which, accepting Walden's value $[M]_D = +270°$, for the bromocamphorsulphonic acid radicle, allows $[M]_D = +48°$ for the basic radicle; the value, $[M]_D = +45°$, previously obtained from the dextrocamphorsulphonate is thus confirmed.

A crucial test of the truth of the explanation based on progressive racemisation is now possible, for if the intramolecular mobility of the base, $(CH_3)(C_2H_5)(C_3H_7)SnOH$, is such that racemisation can effect total conversion of the externally compensated base into the dextrorotatory component, the reverse should occur under favourable conditions. A 1 per cent aqueous solution of the crystalline dextrobromocamphorsulphonate was made in the cold, and the molecular rotation of the dissolved salt was found to be +318°. The solution was then sealed up in a tube and heated on the water-bath for two hours; after cooling, the molecular rotation was found to be now only +273°. As the molecular rotation of the acid is +270°, the base must exist wholly racemised in the solution. Some of the solution was then evaporated to dryness on the water-bath, and, after cooling, made up to its original volume by dissolving the residue in cold water; the molecular rotation of the salt in this solution had risen to +315°. This experiment was repeated with similar results. Further, the addition of potassium iodide solution to aqueous solutions of dextromethylethylpropyl tin dextrobromocamphorsulphonate before and after heating on the water-bath caused the deposition of dextrorotatory and of optically inactive methylethylpropyl tin iodide respectively.

These facts afford the complete proof that on evaporating externally compensated methylethylpropyl tin hydroxide with either of the optically active acids, the lævo-base is wholly converted into the dextrocomponent by progressive racemisation.

Discussion.

Prof. PERCY FRANKLAND called attention to the remarkable mobility of the groups attached to the atom of tin which was quite without parallel in the case of groups attached to carbon. In no asymmetric carbon compound with which he was acquainted had anything comparable to this facility of racemisation been observed.

74. "*Racemic and Optically Active forms of Isoamarine.*" By H. LLOYD SNAPE, D.Sc., Ph.D.

The author has examined the behaviour of the isoamarine previously described by Brooke and himself (*Trans.*, 1899, lxxv., 208) towards nitrous acid, alkyl iodides, acetyl chloride, and nitric acid. Each of these agents acts upon ordinary amarine: and it was desired to contrast the products obtained from the two bases. But, although isoamarine entered readily into reaction in every case, the products were so unstable that as yet the author has failed to prepare them in pure condition.

As the result of further investigation, the anticipation of Japp and Moir (*Trans.*, 1900, lxxvii., 613), that isoamarine is the racemic form of amarine was fully confirmed. Isoamarine, itself optically inactive, was resolved by means of tartaric acid into the acid tartrates,—

$$C_{21}H_{18}N_2{\cdot}C_4H_6O_6{,}2H_2O,$$

of the *d*- and *l*-bases, the salt of the *d*-base being much less soluble than that of the *l*-isomeride. From the respective tartrates, the corresponding bases were obtained by precipitation with ammonia. The *l*-base was more difficult to obtain in a state of purity; but it was evident that the optical antipodes had been obtained, the one giving for $[\alpha]_D$ +62·02°, and the other −61·30° to −58·65°, these values being determined in ethyl acetate solution, after the rotation had become constant.

The racemic base has a higher melting point and density, but a smaller solubility, than the optical antipodes.

Mr. W. J. Pope has kindly measured the crystals of *d*- and *l*-isoamarine, and states that they are orthorhombic prisms, sphenoidally hemihedral, the crystals of the *d*-base showing the form $O\ -k\{111\}$, whilst those of the *l*-isomeride show the form $O\ +k\{111\}$. The crystals of the two substances are thus enantiomorphously related.

The racemic base was synthesised from the *d*- and *l*-isomerides by mixing them in alcoholic solution; but neither of the latter was racemised when heated alone with alcohol to 200°.

PHYSICAL SOCIETY.

Ordinary Meeting, May 25th, 1900.

Prof. J. D. EVERETT, F.R.S., Vice-President, in the Chair.

PROF. S. P. THOMPSON showed some Experiments Illustrating the Aberration called Coma.

If a converging lens is placed obliquely in a parallel beam of light, instead of giving a point image, it produces unilateral distortion, and the bright central spot is accompanied by a pear-shaped tail which is known as a coma. The direction in which this tail points depends upon the side of the lens which is presented to the light. With a concavo-convex lens, the convex surface gives an inward pointing coma, and the concave surface an outward pointing coma. The existence of this phenomenon is due to unequal magnification from different zones of the lens —a fact which was shown by covering the lens with a zone plate of three or four rings, and viewing on a screen the distorted images of the several zones. The form of a coma varies greatly with the distance of the screen from the lens. A parallel beam of light which has passed obliquely through a convex lens, is capable of producing some curious shadows. The shadow of a rod can be obtained as a circular spot, and that of a grating, made by stretching threads between two rods, as concentric circular rings. Prof. Thompson also showed a stringed model illustrating the paths of light rays in the formation of a coma.

Mr. R. T. GLAZEBROOK then read some "*Notes on the Measurement of some Standard Resistances.*"

Three methods have been employed by the author for building up multiples of a standard resistance, such as a 1-ohm coil. The first method consists in making as accurately as possible three 3-ohm coils. These in parallel can be compared directly with the standard by Carey Foster's method. Their resistance in series is very approximately nine times that in parallel, and hence an accurate determination of a resistance about 9 ohms can be obtained. If, then, this resistance is put in series with the standard an accurately known 10-ohm resistance is obtained. By a similar process, a hundred or a thousand ohm coil can be built up. The second method consists in calibrating a resistance box. The 1-ohm coils of the box are compared directly with the standard and the other resistances determined accurately by a building-up process, using a subsidiary resistance box. In comparing the high resistances, the difference between the two boxes may be so great as to send the balance off the bridge-wire. In these cases the third method is employed. The equal arms of the bridge are accurately known, and one of them is shunted with a resistance, which need not be accurately known, until the reading is brought back on to the wire. The coils chiefly used throughout the experiments were made of platinum silver.

Mr. CAMPBELL asked if the same degree of accuracy could be obtained with manganin coils; if so, then the small temperature change of manganin would be an advantage.

Mr. TROTTER asked if proper allowance could be made for the large number of mercury cups used in Method I.

Mr. RENNIE advocated the use of the build-up box in preference to the first method. With two mercury cups there is less chance of errors escaping notice than with eight. Every 10 or 100 ohm coil tested at the Board of Trade is subjected to a comparison with a box calibrated by a build-up process.

Dr. HARKER asked if the resistance of the eight mercury cups in series had ever been measured, and, if so, what was the magnitude of the result and what the uncertainty?

Mr. GLAZEBROOK said he had investigated the resistance of the mercury contacts, and it was negligible. In answer to Mr. Campbell, the author said he had no experience of manganin coils himself, but he had seen some figures for German coils which agreed very closely.

Mr. J. J. GUEST then read a paper "*On the Strength of Ductile Materials under Combined Stresses.*"

The author throughout his experiments has used the "yield-point" of a material as the true criterion of its strength, and has rejected the elastic limit as being modified by local yielding. At present two theories are used in the calculation of strengths of materials. The first is that the material yields when one of the principal stresses reaches a certain amount. This theory, which was adopted by Rankine, and is used by engineers in England and America, is not in accord with recent experiments. The second theory is that the material yields when the greatest strain reaches a certain amount. This was advocated by St. Venant, and is used by engineers on the Continent. Besides these, there is a third theory of elastic strength, in which the condition of yielding is the existence of a shearing stress of a specific amount. In the case of a solid bar subjected to torsion, there is a variation in the strain from the axis outwards, and consequently the materials have been used in the form of thin tubes. This allows the application of an internal fluid pressure. The specimens were of steel, copper, and brass; the state of set caused by drawing having been removed by annealing. The tubes were subjected to—(1) torque, (2) torque and tension, (3) tension only, (4) tension and internal pressure, (5) torsion and internal pressure, and (6) internal pressure only. The axial elongation, the twist, and occasionally the circumferential strain, were measured. Towards the end of the experiments observations were made on bending; the results disprove the maximum stress theory, and are at variance with the maximum strain theory. The maximum shearing stress developed, and the corresponding shearing strain, were comparatively constant throughout the experiments, and no other simple relation between the stresses or strains was even approximately constant. The results of the experiments have been plotted synoptically on a curve, and the several lines have been drawn upon which these points should lie according to the various theories. It is readily seen that the points cluster round the line which represents the existence of a specific shearing stress; the author therefore favours the existence of this stress for any material.

The CHAIRMAN read a communication upon the subject from Dr. CHREE. Mr. Guest, in his paper, has regarded the shearing stress theory as a little known one. As the shearing stress is half the difference between the greatest and least principal stresses, this theory is the same as Prof. G. H. Darwin's maximum stress-difference theory. All the theories suppose that the stress-strain law is linear, and that strains are so small that their squares and products can be neglected. Mr. Guest concludes that, in ordinary materials, the law is linear to the elastic limit, which answers to a stress lower than that which answers to the yield-point, and that yield-point phenomena arise between these. Nevertheless, he focusses attention on the yield point as the criterion of strength, and assumes that Hooke's law holds up to it.

Prof. PERRY congratulated the author upon his paper, and said there was no time left to discuss it at length. The results obtained gave information upon a subject of which very little is known experimentally.

The Society then adjourned until June 8th.

CORRESPONDENCE.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—On p. 678 of "Select Methods in Chemical Analysis" some suggestions are given to avoid bumping in distilling certain fluids. Having occasion to prepare some absolutely pure sulphuric acid (Fresenius's "Qualitative Analysis," §24 A), I introduced (what first came to hand) a few small fragments of ordinary tobacco pipe, and the effect was surprising: the acid distilled away quite calmly, the fluid being covered with bubbles, and the acid dropping regularly into the receiver. I rested the retort (½ litre) upon 1 square inch of asbestos felt resting on a thick clay crucible lid over a large sized Bunsen flame.

I send you this in case you should think the suggestion worthy of mention in the CHEMICAL NEWS, as it may not have been remarked before.—I am, &c.,

G. F. M. FIELDING, M.A.,
Asst. Curate of Staplehurst.

Staplehurst, R.S.O.,
May 24, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. CXXX., No. 19, May 7, 1900.

Preparation of β-Alcoyloxy-α-Cyanocrotonic Ethers, $CH_3—COR=C<^{CN}_{CO_2C_2H_5}$, Isomers of the Aceto-alcoylcyanacetic Ethers, $CH_3CO—C<^{CN}_{R}—CO_2C_2H_5$.—A. Haller.—The silver derivative of acetylcyanacetic ether, when acted upon by alcoholic iodides, causes the formation of those particular ethers whose properties correspond exactly to those of bodies containing an enolic ether function, and not to those whose molecules contain the methinic cetonic function, as the class of acetocyanacetic ethers would lead one to suppose. The presence in these latter of two negative radicles, $CN.CO_2.C_2H_5$, allows of the tautomerism of the molecule, and of changing the cetonic group into an enolic group with the conditions under which the author worked. The results of the experiments lead naturally to the question as to whether acetocyanacetic ether and its analogues really exist in the free state under the form $R.CO.CH<^{CN}_{CO_2.C_2H_5}$, justified by their synthesis, or whether, however prepared, they do not allow of a molecular transposition which makes the β-oxy-α-cyanocrotonic ethers. This property can only be solved by physical methods which the author proposes to investigate.

Heat of Neutralisation of Hydrogen Peroxide by Lime.—M. de Forcrand.—The author continues his research on the subject of the action of oxides on peroxide of hydrogen by substituting lime for the baryta used in his former experiments. The lime, when used in dilute solution, forms with hydrogen peroxide two successive

compounds, the one very rich in oxygen, which has not been isolated, an unstable peroxide or perhaps a compound, $CaO_2+9H_2O_2$: the other, which may be either $CaO.2H_2O_2$ or $CaO_2.H_2O_2$, or possibly $CaO_3.nH_2O$. This latter is analogous to the known compound $BaO_2.H_2O_2$. Calorimetric numbers are given for the various proportions used of CaO and H_2O_2.

Solubility of a Mixture of Salts having a Common Ion.—Charles Touren.—The author performs his experiments under the same conditions as those used for his preceding research on the nitrate and chloride and the nitrate and bromide of potassium. He finds (1) the solubility of potassium chloride in solution of the bromide increasing in concentration, and (2) the solubility of the solid bromide in solutions of the chloride. The study of the solubility of a mixture of salts allows of the recognition of isomorphous salts, when direct proofs are difficult to find.

Action of Phenyl Isocyanate and Aniline on some γ-Cetonic Acids.—T. Klobb.—The author draws the following conclusions from the investigation:—(1) The so-called γ-cetonic acids are transformed under the influence of phenylisocyanate into acid anhydrides, anilides, and pyrrolones. (2) When the acid anhydride is an olide, it does not finally react on the carbanilide, as the acyclic anhydrides studied by M. Haller, it is necessary therefore to substitute aniline. (3) When acids contain two non-substituted groups, CH_2, there are usually formed coloured polymers of pyrrolones, or, more exactly, bodies which are derived by the subtraction of two molecules of water from two molecules of anilide.

New Halogen Mercuric Derivatives of Antipyrine.—J. Ville and Ch. Astre.—In a preceding paper, the authors described a new chlor-mercuric compound of antipyrine corresponding to the formula $(C_{11}H_{12}N_2O)_2HgCl_2.H.Cl$. Under analogous experimental conditions, they have obtained the corresponding bromine and iodine compounds.

Acetyl-phenyl Acetylene and Benzoyl-phenyl Acetylene.—Ch. Moureu and R. Delange.—Under the influence of boiling alkalis, acetyl-phenyl acetylene, a ketone with acetylenic function, $C_6H_5—C\equiv C—CO—CH_3$, becomes split up into acetic acid, $CH_3—CO_2H$, and phenyl-acetylene, $C_6H_5—C\equiv CH$; whilst benzoyl-phenyl acetylene, another acetylenic ketone, $C_6H_5—C\equiv C—CO—C_6H_5$, becomes split up into benzoic acid, $C_6H_5—CO_2H$, and acetophenone, $C_6H_5—CO—CH_3$.

Stability of Solutions of Saccharose.—Œchsner de Coninck.—The author finds that an aqueous solution of saccharose, carefully kept from solar light and the action of mould, at ordinary temperatures is relatively very stable, even in the presence of slight oxidising influences. As soon as moulds begin to form, two sets of transformations take place—first inversion, and then fermentation—producing carbonic acid gas and ethylic alcohol.

Study of the Hydrolysis of Fibrous Tissue.—A. Etard.—The hydrolysis by sulphuric acid of the fibrous tissue of beef causes the formation of a polysaccharide, but does not show any notable quantity of leucine. Glycoprotein is not generally formed, $C_nH_{2n+1}.NO_2+C_nH_{2n-1}.NO_2=C_nH_{2n}.N_2O_4$. All the oxygen is not evolved in the form of carbonyl; a part of the tissues from which it is derived are formed into amino-saccharides.

MISCELLANEOUS.

Research on the Ortho-plumbates of the Alkaline Earths.—G. Kassner.—The author first establishes the fact that the meta-plumbate of calcium, obtained by him by heating the crystalline ortho-plumbate, $Ca_2PbO_4.4H_2O$, and by Grützner and Höhnel by treating Ca_2PbO_4 with Na_2O_2, or the ortho-plumbate with a solution of KOH, contains $4H_2O$, answers to the formula $CaPbO_3+4H_2O$, and only loses three-quarters of its water at 300°. When heated in a current of pure air it gives a more oxygenated plumbic compound. This new compound is a true peroxide having an acid character; this would be perplumbic acid, of which the anhydride should be Pb_2O_5; as this latter is the true peroxide the compound PbO_2 should be called peroxide no longer, but binoxide.—*Archiv der Pharm.*, 237, p. 409.

Purification of Acetylene.—M. Pfeiffer.—At the acetylene works which supplies this gas to the Hungarian State Railways chloride of lime was formerly employed as the purifying material. They now use a mixture of chloride of lime and plumbate of soda containing an excess of alkali. Chloride of lime alone is likely to cause the explosion of the acetylene on account of the liberation of chlorine. A purifier charged with the new mixture was opened after ten hours working. When the upper grating, covered with lime, was removed, spontaneous combustion took place, and a long flame rose from the apparatus, but there was no explosion. Under the above conditions therefore we may say that the new mixture is not dangerous.—*Journ. f. Gasbeleucht.*, lxii., p. 551.

MEETINGS FOR THE WEEK.

TUESDAY, 5th.—Royal Institution, 3. "Ruskin, the Servant of Art," by R. Warwick Bond, M.A.

THURSDAY, 7th.—Royal Institution, 3. "Chaucer," by the Rev. Canon Ainger, M.A., LL.D.

—— Chemical, 8. "Diphenyl- and Dialphyl-ethylene-diamines—their Nitro-derivatives, Nitrates, and Mercurochlorides," by W. S. Mills, M.A. "Condensation of Ethyl Acetylenedicarboxylate with Bases and β-Ketonic Esters," by S. Ruhemann, Ph.D., and H. E. Stapleton, B.A. "The Constitution of Pilocarpine," by H. A. D. Jowett, D.Sc. "The Nitrogen Chlorides derivable from *m*-Chloroacetanilide, and their Transformations," by F. D. Chattaway, D.Sc., K. J. P. Orton, Ph.D., and W. H. Hurtley. "Derivatives of Cyanocamphor and Homocamphoronic Acid," by A. Lapworth, D.Sc.

FRIDAY, 8th.—Royal Institution, 9. "The Effect of Physical Agents on Bacterial Life," by Allan Macfadyen, M.D., &c.

—— Physical, 5. "On the Magnetic Properties of Iron and Aluminium Alloys" (Part II.), by Dr. S. W. Richardson. "Note on Crystallisation produced in Solid Metal by Pressure," by W. Campbell. "On the Viscosity of Mixtures of Liquids and of Solutions," by Dr. C. H. Lees.

SATURDAY, 9th.—Royal Institution, 3. "The Growth of Chamber Music" (with Musical Illustrations), by Sir Frederick Bridge, Mus. Doc.

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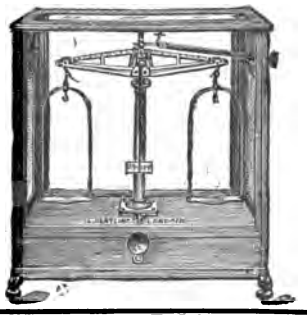

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RADIO-ACTIVITY OF URANIUM.*

By Sir WILLIAM CROOKES, F.R.S.

(Concluded from p. 255).

18. In making photographic tests it is not necessary to take much of the substance. On an ordinary microscopic slide I put a small drop of liquid from each of the highest five fractionations, Nos. 10, 9, 8, 7, and 6. The drops were allowed to crystallise and the slide was laid on a sensitive plate. In twenty-four hours a good impression of No. 10, the highest fraction, was obtained; a less strong impression of the next, No. 9; a fainter one of 8; a scarcely perceptible one of 7; and no impression at all from No. 6. The slide containing the five crystalline spots was then covered with another glass, and the whole cemented together with Canada balsam and mounted in the manner usual with microscopic slides. When the balsam was dry the slide was put on a sensitive plate. In twenty-four hours a good graduated image was developed.

19. There are in commerce two kinds of uranium nitrate: one, the commercial variety, and another called "purissimum." I am informed that the "purissimum" is prepared from the former by repeated crystallisation. I purchased some of each of these nitrates and tested them on a photographic plate. The commercial variety proved to be at least twice as radio active as the "purissimum" salt.

20. Experiments were now instituted with a view of obtaining a wholly inactive uranium nitrate. About two pounds of the salt that had been obtained from the solution in ether was repeatedly crystallised, pouring off the mother-liquor each time. This and the next succeeding two lots of crystals (Nos. 1, 2, and 3) were put into cells, and kept on a sensitive plate for seven days. On developing the plate no image could be detected where No. 1 had been; a scarcely perceptible impression could be just detected at No. 2, and a little stronger impression at No. 3.

21. Other methods were attempted for the separation of the active substance from uranium. Uranium nitrate fuses at a moderate temperature, and after some time it becomes darker, and nitrous fumes come off. Finally, the mass becomes semi-fluid, and will not run. The operation is then stopped, and the mass transferred to water; the undecomposed nitrate is dissolved out, leaving an insoluble basic nitrate. The basic nitrate is of an orange-yellow colour, easily dissolved in nitric acid to again form the normal nitrate. By using this method of fractionation the active body gradually accumulates towards the basic end, but the method is neither so complete nor so easily effected as the crystallisation method, and therefore I have not pushed it very far. I have, however, proved that the radio-activity of nitrate of uranium can be concentrated by fractionation to the basic nitrate end, the nitrate at the other end being diminished in radio-activity.

22. A highly active uranium nitrate, prepared by fractionation from the part insoluble in ether, was dissolved in water, and ammonia in excess was added. Yellow ammonium uranate was precipitated. The filtrate was evaporated to dryness, and heated with nitric acid. The yellow precipitate and the residue of the filtrate were put into cells, and laid on a sensitive plate. After twenty-four hours' action the plate was developed, when it was seen that the whole of the radio-activity resided in the ammonium uranate, the other substance showing nothing This experiment proves that the active body is precipitated by ammonia, and is insoluble in excess.

23. Another portion of active uranium nitrate was dissolved in water, with an excess of ammonium carbonate. The first-formed precipitate almost entirely re-dissolved, leaving a small quantity of insoluble light brown flocculent precipitate. This collected on warming, like alumina. It was filtered off, well washed and dried, and put in a glass cell.

The filtrate from the above precipitate was evaporated to drive off the ammonium carbonate, when a yellow precipitate came down. This was filtered, washed, dried, and put into a cell.

These precipitates were exposed for twenty-four hours in a lead screen apparatus. On developing, it was seen that the residue insoluble in ammonium carbonate instantly flashed out black and dense, while the salt precipitated from the ammonium carbonate solution gave a scarcely discernible image.

24. The action of the precipitate insoluble in ammonium carbonate was so strong that another experiment was tried, exposing the sensitive plate to its action for one hour. On development, the disc of action came out strong and black, although not so black as in the twenty-four hours' experiment. It was now laid for five minutes on a sensitive plate. Here the action was distinct—about as strong as that given by ordinary uranium nitrate in twenty-four hours. These experiments prove that the active body can exist apart from uranium.

If a sheet of thin glass or celluloid is laid on a sensitive plate, and the dried filter-paper with its contents laid on that, and kept down by a weight, an impression is given in as short a space of time as if a glass cell had been used.

25. The radio-active body is not entirely insoluble in ammonium carbonate. A portion of the very active precipitate left after separation from the uranium was dissolved in dilute hydrochloric acid, and an excess of ammonium carbonate added. The precipitate was very brown, due to the presence of iron; it was dried and tested. The filtrate was well boiled, and as the ammonium carbonate evaporated a slight precipitate came down. This was collected on a filter and tested by the side of the first precipitate. On developing the plate the images produced by each precipitate were of about equal intensity. The brown precipitate was digested in very dilute hydrochloric acid in the cold. The iron partially dissolved before the rest of the substance, leaving the residue decidedly paler in colour. This pale body was just as radio-active as before the partial removal of the iron. Therefore the presence of iron does not interfere with the activity of the substance.

26. Having thus definitely proved that the supposed radio-activity of uranium and its salts is not an inherent property of the element, but is due to the presence of a foreign body,* it is necessary patiently to determine the nature of the foreign body. Several radio-active bodies claimed to be new have already been extracted from pitchblende, and experiments have been instituted to see if the newly found body UrX had similar chemical properties to those of the older active substances.

27. Polonium was first tried. A photographic plate had a thin sheet of celluloid laid on it, and over this a sheet of aluminium foil, 0·05 m.m. thick. On this double layer were put two cells, one containing basic polonium nitrate, the other active UrX. Action was allowed to proceed for twenty-four hours, and the plate was then developed. A disc of blackening was seen under where the UrX stood, the action having passed through the glass, celluloid, and aluminium. Under the polonium nitrate no trace of action could be detected.

The experiment was repeated, minus the aluminium foil, and the action continued only two and a quarter

* A Paper read before the Royal Society, May 10, 1900.

* For the sake of lucidity the new body must have a name. Until it is more tractable I will call it provisionally UrX—the unknown substance in uranium.

hours. On development, the UrX was found to have acted well, while the polonium showed no trace of action.

28. This behaviour of polonium being eccentric or contrary to published accounts,* I put some polonium nitrate in a very thin gelatin capsule, and laid it for eight hours on a sensitive plate. No trace of an image could be seen on development.

The same polonium nitrate was put in a watch-glass, and the sensitive plate put *over it* face downwards, so that it might be exposed to the direct emanations from the polonium nitrate. On the top of the plate was laid a sheet of lead to press it tight to the edges of the watch-glass.

The exposure was continued for twenty-eight hours. On developing, a strong action was seen, strongest in the middle where opposed to the thickest part of the heap of polonium nitrate, and weaker towards the edge. A well-marked action took place all over the plate exposed to the interior of the watch-glass, but it was sharply cut off at the edges. This confirms the previous results—that the emanations from polonium are of a different character to those from radium or UrX, both of which pass through glass, aluminium, and lead.

29. Another property of polonium sharply distinguishing it from UrX is volatility. The discoverers first obtained it by subliming pitchblende *in vacuo*. Afterwards they used this property to separate it from bismuth, the polonium and the bismuth sulphides depositing at different parts of the hot tube.

A strongly radio-active compound of UrX was ignited in a blowpipe flame with the addition of a drop of sulphuric acid. Its radio-activity, on a sensitive plate, was not diminished by this treatment. This experiment was tried several times at increasingly higher temperatures, and always with the same result.

30. Polonium is precipitated by sulphuretted hydrogen, in an acid solution. An acid or neutral solution of UrX is not precipitated by this reagent. Therefore I am justified in saying my UrX is not polonium.

31. But it is not so easy to settle whether UrX is distinct from radium, although many arguments point to its not being radium. The discoverers of radium give several of its chemical properties, and in most of these UrX and Ra are entirely different. Thus, radium sulphate is said to be insoluble in water and acids, while UrX dissolves easily to a clear solution in dilute sulphuric acid. Radium salts are said to be not precipitated by ammonium sulphide or by ammonia, while UrX is precipitated by both.

32. It was hoped that doubtful points might be settled conclusively by the spectrum, as both radium and polonium give well-defined and characteristic lines, especially in the ultra-violet part of the spectrum where I have chiefly worked. M. Demarçay (*Comptes Rendus*, vol. cxxiv., p. 716; Chem. News, vol. lxxx., p. 259) has given a list of some of the principal lines in the radium spectrum between the wave-lengths 3649·6 to 4826·3, the one at 3814·7 being very strong, and those at 4683·0, 4340·6, and 3649·6 being next in intensity. He draws special attention to the line at 3814·7 as the line showing first in a compound poor in radium. In none of my UrX compounds have I been able to detect a trace of this line; on the other hand I have failed to photograph this line in products which I know contain radium. The reason is my radium compound is too weak. M. Demarçay says the line is scarcely visible with a radium compound only sixty times as active as uranium. My substance containing radium was still weaker, judging from its action on a photographic plate.

33. The same reasoning applies to polonium. With polonium I have obtained strong lines in the ultra-violet, but I can detect none of them in the spectrum of my compound of UrX. All that I can see are lines belonging to—

Platinum (from the poles),
Uranium,
Calcium,
Aluminum,

and a few of the strongest air-lines, besides a large number of faint lines difficult to identify.

34. Spectrum experiments having failed to show a difference between radium and UrX, it was thought that possibly some information might be gained by submitting them to the radiant matter test, which has proved so fruitful in its application to the yttrium earths. Some of the most active UrX was put in a tube furnished with a pair of terminals, and it was exhausted to a high point, heat being applied during exhaustion. Simultaneously a self-luminous radium compound was sealed in a vacuum tube and exhausted, heat being likewise applied. When fully exhausted a strong induction spark was passed through each tube. The UrX compound phosphoresced of a fine blue colour. In the spectroscope no discontinuity could be seen in the spectrum of the phosphorescent light.

Under the influence of the induction spark, the radium compound phosphoresced of a luminous rose-colour, showing in the spectroscope a concentration of light in the red-orange, and a very faint citron band, due to a trace of yttrium, probably an impurity.

35. A powerful radium compound and one of UrX, each in a glass cell, and a paper tray full of polonium subnitrate, were placed side by side, and a strip of white card was put as a reflector at the back. In front a photographic camera was arranged so as to throw full-sized images of the polonium, UrX, and radium compounds on a sensitive plate, and the whole was kept in total darkness for five days. On development, the image of the radium with the containing bottle was visible, but not a trace of image from the polonium could be seen. This confirms previous observations that the radiations from polonium will not pass through glass. Those from radium and UrX easily penetrate glass and other media (28).

36. Recently claims have been put forward for the existence of a third radio-active body in pitchblende. In the *Comptes Rendus* for October 16, 1899, and April 2, 1900, M. A. Debierne describes a radio-active body, to which he gives the name of "Actinium." At first he said "actinium" showed the principal analytical properties of titanium, but later he describes it as not resembling titanium in all its reactions. M. Debierne gives many reactions of the new substance, and in some instances they are like those of radium. But he qualifies them by the statement that they cannot yet be considered as belonging definitely to the new radio-active substance, because up to the present it has not been obtained sufficiently concentrated. He believes rather that these reactions should be looked upon as the result of retention, analogous to that of iron oxide by barium sulphate. He says that the chemical reactions of the most active substance which he obtained, together with its spectroscopic examination, showed that it chiefly consists of thorium, He cannot, however, be sure that it resembles thorium in all its reactions.

37. Experiments have been commenced to see if it is possible to separate thorium compounds into an active and an inactive body. A strong solution of thorium

* "The rays emitted by compounds of polonium render barium platinocyanide fluorescent. . . . To make the experiment, place on the active substance a very thin sheet of aluminium, and on this a thin layer of barium platinocyanide; in the dark the barium platinocyanide appears feebly luminous over the active substance" (M. and Mdme. Curie and M. Bémont, *Comptes Rendus*, vol. cxxvii., p. 1215; Chem. News, vol. lxxix., p. 1). "Polonic rays act on sensitive plates. The substance we call sulphide of polonium gives a good impression after only three minutes, and there is a decided action noticed after even half a minute" (Mdme. Curie, *Revue Générale des Sci.*, January 30, 1899; Chem. News, vol. lxxix., p. 77). In the same paper the authoress, after describing the power possessed by a polonium compound to excite phosphorescence, says, "the rays emitted by this latter body have traversed the aluminium and excited the fluorescence of the platinocyanide above it." It is evident from the above extracts that I was justified in thinking that polonium rays were not entirely stopped by thin aluminium, glass, or celluloid.

sulphate was slightly acidulated with sulphuric acid, and gradually raised to the boiling point. A copious precipitate of sulphate came down, and was filtered hot. The precipitate was dissolved in cold water, and the solution re-heated, when a precipitation of the sulphate again occurred. The mother-liquor from one crystallisation was added to the crystals from another in the systematic manner adopted in fractionation, and when the operations had proceeded some time a test was made on the "head" and "tail." A small quantity of solution from each was evaporated to dryness and strongly ignited before the blowpipe. The two lots of earth were put in cells and a sensitive plate exposed to their action for seventy-two hours. On development not the slightest difference could be detected between the impressions produced by either of the fractions.

I next tried partial crystallisation of thorium nitrate, fractionating it in the way already described in the case of uranium (15). Great difficulties were here encountered, owing to the tendency of a strong solution of thorium nitrate to remain supersaturated for several days, when it would suddenly crystallise to a solid mass. After some weeks, however, six fractionations were effected, and tests were made on the first and last of the series.

The sensitive plate was exposed to their action for 120 hours. On development, the fraction at the first end (crystals) gave a very feeble action, while that at the other end (mother-liquors) gave an impression about three times as intense. This points to the possibility of separating from thorium its radio-active substance.

By the kindness of Dr. Knöfler, of Berlin, who makes thorium nitrate by the ton, I have at my disposal some specially prepared thorium nitrate, which is chemically pure. Thoria prepared from this, tested on a sensitive plate, gave a feeble impression in 120 hours.

38. In the present state of our knowledge of these radio-active bodies it is safest to retain an open, or even a slightly sceptical mind. We recognise them mainly by the photographic and the electrical tests—reactions which are so sensitive that they give strong results, even when the active body is present in too small a quantity to be detected by its spectrum—one of the most delicate of tests. Knowing the tendency of ordinary chemical bodies to be carried down when a precipitate is formed in their presence, even when no question of sparse solubility is involved, it is not surprising that radium and actinium, to say nothing of UrX, appear to simulate elements which may ultimately prove to be very different from them in chemical characters. For instance, UrX dissolves easily in dilute sulphuric acid, and, I have reason to believe, forms a soluble sulphate; still, when chloride of barium is mixed with it and precipitated as a sulphate, I invariably find strong radio-activity in the precipitated sulphate as well as in the filtrate from the barium sulphate.

To adduce a simile from my previous researches, the first surmises as to the chemical characteristics of the bodies now known to be yttrium and samarium, were widely different from reality. The differences were entirely due to the perturbing cause which is active in the present case—the tendency of the bodies to be carried down and entangled in precipitates, where, according to ordinary chemical laws, they ought not to occur; and to the extreme delicacy of the radiant matter test, which in the case of samarium detects one part in 2½ million parts of calcium, and in the case of yttrium detects one part in the presence of a million parts of extraneous matter.

39. The radiographic test for these active bodies presents another point to be borne in mind. Other tests for the presence of an element either act quickly, or do not act at all, with a comparatively narrow margin of debateable land where the indications of the test may be doubtful. Here, however, the test is cumulative. Like an astronomer photographing stars too faint for his telescope to disclose, he has only to expose the plate for a sufficiently long time and the star reveals itself on development. So, in the case of radio-active minerals or precipitates, if no action is apparent at the end of an hour, one may be shown after twenty-four hours. If a day's exposure will will show nothing, try a week's. Considering my most active UrX does not contain sufficient of the real material to show in the spectrograph, yet is powerful enough to give a good impression on a photographic plate in five minutes, what must be its dilution in compounds which require an hour, a day, or a week to give an action?

ON THE PHOTOGRAPHY OF THE SMALLEST WAVE-LENGTHS OF LIGHT RAYS.*

By VICTOR SCHUMANN, Leipzig.

My observation of the spectrum beyond the wave-length 185 $\mu\mu$—which work I have resumed after several years' interruption—led to the following results:—

1. The photographic examination of this region of the spectrum now succeeds in much less time than formerly, in consequence of repeated improvement of my means of observation. The hydrogen lines at 100 $\mu\mu$ require as the result only a few seconds' exposure.

2. The transparency of quartz diminishes appreciably with the wave-length. A plate of quartz of 0·5 m.m. thickness absorbs nearly all rays beyond 150 $\mu\mu$.

3. White fluor-spar behaves similarly, but up to 100 $\mu\mu$ is much more transparent than quartz, but it weakens in a striking degree all the more refrangible rays. For example, in only 0·5 m.m. thickness, it more than doubles the time of exposure. This loss of light depends less on the thickness of the fluor-spar than on the number of its reflecting surfaces which the rays pass through.

4. Hydrogen in this region possesses a second line spectrum; it appears at atmospheric pressure, and only with the spectrum caused by discharge between the electrodes. It strongly resembles the low pressure spectrum, but shows also rays of other wave-lengths and a somewhat different distribution of energy. The production of the metallic spectra is complicated by the simultaneous appearance of the lines of this spectrum, so that the ascertainment of the origin of individual lines still presents difficulties.

5. Metallic spectra require much longer illumination than the low pressure spectrum of hydrogen. Firstly, because the metallic spark produced in an atmosphere of hydrogen acts materially weaker than in air; and, secondly, because—from above-mentioned reasons—a considerable portion of the active light is lost in the fluor-spar window separating the discharge area from the vacuum spectrograph.

THE TRANSPARENCY OF THIN FILMS OF GLYCERIN.

By Dr. VICTOR SCHUMANN.

The use of glycerin for joining together the halves of the Cornu quartz prisms induced me to determine the absorption relations of this medium, especially towards the most refrangible aluminium rays. The result thus obtained, in consideration of the frequent use of these prisms in spectrum photography, is so far important that glycerin is not so indifferent towards the above-mentioned regions of the spectrum as it has proved to be in the less refrangible ultra-violet, according to the observations of Eder and Valenta.

* Read before the Imperial Academy of Sciences in Vienna, March 29th, 1900.

According to my experience, two quartz plates, cut perpendicularly to the axis, containing a glycerin film 0·1 m.m. thick, increased the times of exposure of the aluminium rays—

No. 30, λ 199 $\mu\mu$, from 18·0 to 25·0
No. 31, λ 194 $\mu\mu$, from 11·4 to 25·0
No. 32, λ 185 $\mu\mu$, from 0·08 to 25·0

When I afterwards stuck the two quartz plates together with a very small quantity of glycerin, to prevent reflection at the two inside surfaces and to obtain the thinnest film possible, the times of exposure on repetition of the experiment were considerably less increased, viz. :—

No. 30 and No. 31, from 19·5 to 25·0
No. 32, from 17·5 to 25·0

These numbers prove how much the transparency of a double quartz prism depends on the thickness of its glycerin film, and that the unobstructed passage of the most refrangible rays is only possible with an extremely thin layer of glycerin. That this combining medium should be preferred in certain circumstances to the dry contact of the prism halves (provided that the glycerin film is not much thicker than in the last-mentioned case) can be concluded from the values obtained when the quartz plates are laid dry upon one another. The dry plates lengthened the time of exposure of the rays—

No. 30 and No. 31, from 16·8 to 25·0
No. 32, from 15·0 to 25·0

From all these values, it is evident that the most refrangible rays must be carefully dealt with in order to obtain their intensity undiminished.

With regard to the less refrangible ultra-violet downwards to wave-length 227 $\mu\mu$, I may mention that I was not able to detect with certainty any influence of the glycerin film 0·1 m.m. thick. It is possible that a want of sensitiveness in my instruments, not specially adapted to photometric work, is responsible for this negative result. But it must not be said that the absorption vessel with its contents did not affect the photographic action. On the contrary, it lengthened—in consequence of loss of light resulting from reflection—the time of exposure for the region from 361 $\mu\mu$ to 227 $\mu\mu$ almost uniformly from 21·5 to 25·0. As for the more refracted rays, which gave evidence of the influence of the glycerin, the time of exposure—for example, at Cd No. 25, λ 220 $\mu\mu$, and at Cd No. 26, λ 215 $\mu\mu$—rose from 19·5 to 25·0, whilst under otherwise similar conditions the glycerin vessel alone caused a rise from 21·5 to 25·0 only.

NOTE ON THE PECULIAR DIFFICULTIES WHICH BESET THE APPLICATION OF THE AMMONIA METHOD TO THE ANALYSIS OF SEWAGE AND SEWAGE EFFLUENTS.

By J. ALFRED WANKLYN.

In our treatise on "Sewage Analysis," published this day (May 12th), last year, my colleague, Cooper, and myself, remarked as follows :—

"The ascertainment of the true figure for the albumenoid ammonia in sewage and sewage effluents involves some practical difficulties, and we fear that much of the work put forward by our chemical brethren is invalid. We suspect that serious errors, both in the direction of an over-estimate and an under-estimate, have been made on many occasions when sanitary officials have employed the ammonia method in the examination of sewages and sewage effluents."

Within the last few months I have had very striking evidence of the necessity of the warning which I have just quoted.

The following is a characteristic example of the serious analytical divergencies which occur when the ammonia method is applied to very similar and presumably identical sewage effluents by two independent analysts.

Results obtained by analyst A. :—

Sewage effluent	No. 1	gave	1·00	albumenoid	ammonia.
,, ,,	No. 2	,,	0·80	,,	,,
,, ,,	No. 3	,,	1·10	,,	,,
,, ,,	No. 4	,,	0·80	,,	,,
,, ,,	No. 5	,,	0·80	,,	,,
,, ,,	No. 6	,,	0·90	,,	,,
,, ,,	No. 7	,,	0·90	,,	,,
,, ,,	No. 8	,,	0·90	,,	,,
,, ,,	No. 9	,,	0·80	,,	,,

Results obtained by analyst B. :—

Sewage effluent	No. 1	gave	3·80	albumenoid	ammonia.
,, ,,	No. 2	,,	6·85	,,	,,
,, ,,	No. 3	,,	13·50	,,	,,
,, ,,	No. 4	,,	8 00	,,	,,
,, ,,	No. 5	,,	8·50	,,	,,

The practical effect of the two series of analyses being that, whereas the analyses by analyst A. showed that the effluent was very good, the analyses by analyst B. showed that the effluent was utterly bad.

The two sorts of effluents were not of quite the same date, but they were effluents from the same sewage works.

If not quite identical in composition, they were doubtless very similar in character and composition; and, if correctly carried out, the analysis of the two sets of effluents would, I believe, have exhibited that similarity.

I am personally acquainted with analyst A., and know that he adopted special precautions to avoid the mistake of returning ammonia which had been derived from urea as if it had been derived from substances coming under the category of the albumenoid group of substances.

With analyst B. I have no personal acquaintance whatever. Apparently the gentleman is personally known to, and enjoys the confidence of, one of the lecturers on Public Health, whose knowledge of the analysis of sewage by the ammonia method appears to have been derived from my treatise on "Water Analysis" before the publication of the "Sewage Analysis" last year.

In reference to the description of the analysis of sewage given in my treatise on "Water Analysis," I have to say that that description was, of course, only supplementary to the main subject of that book, and that it was very much abridged, and, unless read with more intelligence and care and discrimination than might fairly be demanded, it might have led the analyst into error.

One of the greatest difficulties of sewage analysis—perhaps I should say the chief difficulty of sewage analysis—depends upon the extraordinary properties of urea.

Urea is very prone to undergo transformation into carbonate of ammonia by reaction on some of the water in which it is dissolved.

In many sewages, the great bulk of the urea originally present in the urine has undergone this change before the sewage leaves the sewers. But there are great differences in this respect in different sewages. Sometimes fully half of the original urea remains undecomposed in the sewage as it leaves the sewer. In such cases an analyst working the ammonia method in a somewhat perfunctory manner—laboriously, but not discreetly—and without the special information to be found in our recent book on sewage-analysis, might be expected to obtain results similar to those found by analyst B. The following experiments, recently made in my laboratory, are instructive and much to the point :—

Fresh urine, sp. gr. 1018·4, was mixed with good London water from the Lambeth Water Company in the proportion of one volume of urine to 99 volumes of water. A considerable quantity of this dilute solution of urine was prepared for the purpose of experiment.

The same day, whilst it was fresh, this dilute urine was

submitted to analysis in the "somewhat perfunctory manner—laboriously, but not discreetly"—and without regard to the special information contained in the treatise on "Sewage Analysis." It yielded:—

	Milligrms. per litre.
Free ammonia	12·50
Albumenoid ammonia	49·00

In this case the dilute urine, representing sewage or sewage effluent, was almost quite unfermented, and the old and well-known but seldom worked method of chemical transformation of the unfermented urea into carbonate of ammonia was not resorted to. The correct figure for the albumenoid ammonia instead of being 49·00 milligrms. per litre was only about 6·00 when the method was properly worked.

After keeping the dilute urine for ten days at the ordinary temperature of the atmosphere, it was again analysed, and gave totally different results, owing to the circumstance that after that length of time the fermentation of the urea into carbonate of ammonia had run its course. The results were as follows:—

	Milligrms. per litre.
Free ammonia	100·00
Albumenoid ammonia	3·50

The point to which I desire to direct attention is that no return of the albumenoid ammonia in sewage or sewage effluent is trustworthy unless adequate precautions have been taken to avoid mistaking urea for the complex nitrogenous organic substances which yield albumenoid ammonia.

The Laboratory, New Malden.

THE ESTIMATION OF MOLYBDENUM IN STEEL AND STEEL-MAKING ALLOYS.

By FRED IBBOTSON and HARRY BREARLEY.

It is no easy matter to arrive at a satisfactory conclusion from published opinions as to the influence which the presence of molybdenum exerts on steel. Von Lipin (*Stahl und Eisen*, xvii., 571), points out that such steels need very careful heat treatment during the working of them; that after hammering at a heat not exceeding a cherry-red, they are quite clean and free from the small cracks which are often present in tungsten steels; that compared with a steel containing an equal amount of tungsten, one containing molybdenum is softer, that is, gives greater elongation; that the results of water hardening are more, and of oil hardening less marked; and that molybdenum steel will bear more mechanical punishment without de-developing cracks. Experiments at the Creusot Works showed that in the presence of chromium even a few tenths per cent of molybdenum were advantageous in the manufacture of armour plates. Amongst other particular properties of molybdenum Moissan and others have pointed out that it would prove valuable as a deoxidising agent in the converter and elsewhere during the manufacture of steel, as the MoO_3 produced would be quite volatilised.

Mo has been found in the specular iron ore of Sweden by Ingelström, in Belgian coal by Armand Jorissen, in pig-iron by Ledebur, and in blast-furnace refuse by several observers. If we remember that tungsten compounds are rarely free from molybdenum, and that tungsten steel is an important commercial product, it is very likely that molybdenum has been frequently overlooked, particularly as its analytical characters so much resemble those of tungsten.

At present, steel which actually contains notable quantities of Mo is rare; many analysts have never had a sample passed through their hands. A lamentable illustration of this statment is the fact that small amounts of Mo in cast-iron have been mistaken by two English chemists for a new element.

This comparative rarity is doubtless accountable for the fact that no English text-book, so far as we know, devoted to metallurgical analysis, gives instructions for estimating Mo in iron compounds. Carnot has, so we are informed, described a process in the *Annales des Mines*, which we have, however, not seen.

In our earliest experiments we obtained satisfactory results by fusing the oxidised alloy with Na_2CO_3, extracting with water, reducing the filtrate with zinc, and titrating with permanganate. This process was used by Benneville (*Journ. Iron and Steel Inst.*, 1895, i., 202).

In this and a subsequent paper we offer gravimetric methods for the estimation of Mo and W, which are based on the various processes elaborated in our recent papers on the analysis of Mo and W compounds.

Molybdenum in Steel.—It has been shown already (Chem. News, lxxix., 3), that the separation of Fe and Mo increases in accuracy as ammonium acetate, hydrate, sodium carbonate, or sodium hydrate are used, and that with the last reagent in particular absolute accuracy of separation may be obtained when the iron present is only two or three times greater than the Mo. In preparing a series of test analyses to illustrate the value of the method for both high and low percentages, it became very plain that from about 15 per cent of Mo downwards, the separation was inaccurate.

The strong likeness between the behaviour of chromic and molybdic acids in relation to their separation from iron has already been commented on. Still it is important here to re-emphasise the fact that on adding an alkali to an acidified mixture of ferric chloride and molybdic acid, a point is reached at which insoluble basic ferric molybdate is formed, and that no perfect separation of the two metals is possible until sufficient alkali has been added to decompose this compound. For the separation of small amounts of iron this excess need not be a large one, so long as the precipitant be of the right kind; but as the amounts of iron in association with molybdic acid become larger and larger, the same excess of the precipitant becomes less and less able to effect a complete separation. The addition of increasing amounts of soda hydrate improves the separation only very slowly after a certain approximate separation is reached, and no system of aërating the precipitated flocks of ferric hydrate, such as by the use of a mixture of sodium hydrate and carbonate, will effect the desired end.

A description of an entirely satisfactory process applied to a steel or ferro molybdenum of small Mo content is as follows:—

Dissolve a suitable amount (say 2 grms.), of the sample in HCl. The unusual character of the metal is at once indicated by the colour of the solution. Oxidise with HNO_3 or $KClO_3$, add soda carbonate to destroy all or nearly all the free acid, taking care to stop the addition short of the production of a red colour or precipitate. Pass through a small pulp filter, place the filter in a flask containing 30—40 c.c. more soda hydrate of strength 2 N than is necessary to precipitate the whole of the iron. This amount is easily calculated if nearly all the free acid has been neutralised, because 1 grm. of iron as Fe_2Cl_6 would require 54 c.c. normal soda to just precipitate it. There is no exceptional advantage in this particular excess of NaHO except that it is sufficient without being wasteful, and large excesses tend to retard subsequent operations.

After disintegrating the added filter, which may possibly contain a little MoO_3 or WO_3, the contents of the flask are heated nearly to boiling, the flask placed under a funnel with a constricted stem, and violently shaken with one hand, whilst the hot sample is poured through the funnel with the other. The Mo is all in solution, and after fractional filtration and acidification of the clear solution with HCl, the metal is precipitated as $PbMoO_4$ in the manner already described (Chem. News, lxxviii., 203).

The following table shows how perfect the above separation is. The $PbMoO_4$ precipitated from the filtrate was always re-dissolved in HCl, a small quantity of SiO_2 separated, and the Mo re-precipitated. Without this precaution, the first precipitation gave results averaging a plus error of 1 per cent:—

Grms. of iron.	Mo added.	Mo found.
0·2	0·2	0·1998
0·5	0·25	0·2485
0·6	0·15	0·1499
0·9	0·10	0·0998
2·0	0·015	0·0151
2·0	0·0025	0·0026
2·0	0·0005	0·00047

Some experimental samples of cast-iron containing from 3 to 4 per cent of Mo were successfully assayed in the above manner, although it is perhaps better to filter off the graphite, burn the filter at low redness, and fuse the residue with KNO_3. This was advantageous in the particular samples alluded to, because they also contained tungsten in considerable amounts.

The estimation of Mo in steel as carried out by this process is not interfered with by any of the usual elements found in steel, such elements being either precipitated along with the iron or exercising no influence in the subsequent precipitation of the Mo with lead acetate; this remark, of course, does not apply to tungsten. Appended are a few results:—

	Per cent.
Mo found in steel free from Ni and Cr	2·72
Mo found in same steel after adding 20 p. c. Ni..	2·73
,, ,, ,, 20 ,, Cr..	2·72
,, ,, ,, 20 ,, Mn.	2·75

In the case of chromium there was probably a small quantity of chromium in the filtrate as chromate. A few drops of SO_2 solution were added before precipitation. The ammonium acetate which we always add before filtering the boiling mixture effectually holds in solution the small quantity of lead sulphate which might otherwise accompany the $PbMoO_4$.

We think it desirable to mention here the results of experiments instituted to determine whether Mo in steel interferes with the estimation of the other elements, and how its interference may be overcome:—

Silicon.—Molybdenum does not interfere.

Manganese.—If estimated by separation of the iron with ammonium acetate and precipitation with Br and ammonia, Mo exerts no influence. Unless a large excess of acetate is used, nearly all the Mo is precipitated with the iron. In the volumetric determination by oxidation with $KClO_3$ in nitric acid solution, the results are low. A steel with Mn contents of 0·60 per cent gave only 0·39 per cent. By the addition of six instead of three separate lots of chlorate, the full amount of manganese was obtained.

Sulphur.—The gravimetric estimation of sulphur is accurate if the $BaCl_2$ is added to a distinctly acid solution in order to eliminate the possibility of contamination with $BaMoO_4$ or basic ferric molybdates. The evolution method, *i.e.*, the dissolution of the steel in HCl, the evolved H_2S being passed through metallic solutions, is also uninfluenced by the presence of Mo. One would expect low results due to the formation of MoS_3 in the evolution flask. This does not seem to be the case when strong HCl is used to effect decomposition.

Phosphorus.—The long (acetate) molybdate method yields accurate results in the presence of Mo. The rapid method, in which the P is precipitated in the presence of all the iron, gives high results, apparently in consequence of contamination of the yellow precipitate with MoO_3. The difficulty may obviously be overcome by dissolving out in ammonia and precipitating with magnesia mixture.

Carbon.—The carbonaceous residue, after decomposing the sample with copper solutions, gives blue washings (Mo_3O_8) long after the copper salts have been washed out. The results, compared with direct combustions with PbO_2 or Pb_3O_4, are a few hundredths per cent too low.

Iron.—On precipitating with ammonia part of the molybdenum accompanies the iron; gravimetric results are therefore too high. The iron cannot be estimated by simply dissolving the sample and titrating, because the molybdenum, reduced by the liberated hydrogen to Mo_2O_3, falsifies the result by being oxidised to MoO_3. For a like reason ferric solutions containing molybdenum should not be reduced with zinc or stannous chloride. They may, however, be reduced with SO_2, and titrated accurately either with bichromate or permanganate. Potassium ferrocyanide does not certainly indicate ferric iron, on account of the very ready formation of a deep brown precipitate with MoO_3 solutions.

Analysis of Ferro-molybdenum and Fused Molybdenum Metal.—These alloys are readily attacked by acids, and analysed as just described.

Analysis of Nickel molybdenum.—These alloys generally contain very little iron, so that, after decomposing the sample with aqua regia, the solution is poured into an excess of ammonia. If more than a few tenths per cent of iron are present, the precipitate contains small amounts of Ni and Mo, which may be removed by a second precipitation. The Mo may then be precipitated from a portion of the combined filtrates without separating the nickel, and the nickel may be cyanometrically estimated without separating the Mo. Where iron is present in very large amounts, the molybdenum should be separated as for steels, by means of NaHO, and the Ni and Fe separated afterwards in a fresh sample for the estimation of Ni. This latter separation may be effected with the greatest facility by means of ammonia, a process which depends for its success upon the fact that basic ferric molybdate forms when considerably less ammonia is added than would make the solution alkaline.

Nearly every commercial sample of ferro-molybdenum or nickel-molybdenum we have examined contained tungsten amounting to 1 or 2 per cent. A sample of molybdenum-nickel, which was too tough to break and exceedingly difficult to drill, contained 3 per cent of sulphur.

Analysis of Molybdenum Powders.—A comparison of the analysis of these powders as submitted by their vendors, with those representing actual composition, affords the strongest possible evidence of the need of some definite instructions regarding the analysis of them. We have never met with a sample which professed to contain less than 95 per cent of Mo, and have never found one which contained as much as 90 per cent.

An estimation of the separate elements, or combination of elements, which make up a common molybdenum powder, would be a very difficult piece of work, as it may contain, besides metallic Mo, such bodies as MoO_3, mixtures of the lower oxides of Mo, metallic W, oxides of W, silica, alumina, iron, ferric oxide, combined carbon, graphite or charcoal, sulphur and water in quite appreciable quantities. It is certainly desirable, if powder continues to be the form in which this metal is supplied to steel manufacturers, to have more or less ready means of submitting the material to a complete examination. At present we are disposed to make ourselves responsible only for such processes as may be used to determine the more important constituents.

Carbon.—The total carbon may readily be determined by direct combustion in a current of oxygen. Moissan, in a very interesting paper (Chemical News, lxxii., 2), determines the combined carbon by attacking the sample with acids, passing the gases over copper oxide, &c., into KHO. the graphite being determined in the insoluble residue.

Molybdenum.—For the estimation of the chief element the powder may be mixed with Na_2CO_3 and a little KNO_3 to promote oxidation, placed in a platinum crucible under a layer of Na_2CO_3, and heated over the blowpipe until

decomposed. Only the bottom of the crucible should be kept red-hot, so as to avoid volatilisation of any MoO_3. After extracting with water, the Mo and W are precipitated in the filtrate from the insoluble Fe_2O_3, &c., with lead acetate, and separated as described by us (Chemical News, lxxxi., 13).

A more complete examination of the sample is possible if 3 to 5 grms. are opened out with aqua regia, the solution being evaporated dry, and the residue well boiled with concentrated HCl, diluted, and filtered. The residue, after ignition at low redness, may contain WO_3, SiO_2, and MoO_3. It is fused with KNO_3, and the aqueous extract added to the main filtrate or analysed separately. A portion of the main filtrate is used for estimating the Mo and W after separating the small amounts of iron and alumina with ammonia. Another portion may be used for estimating sulphur, and so on.

Oxygen.—The better qualities of powder supplied to steel-makers are grey and anhydrous; the poorer qualities are black, and may contain 5 to 6 per cent of water. Owing to imperfect reduction in the first instance, or subsequent decomposition, assisted perhaps by exposure, molybdenum powders may contain, besides the metal itself, its oxides Mo_2O_3, MoO_2, MoO_3, and Mo_3O_8. We are unable to supply a satisfactory method for the estimation of these oxides separately, and think it hardly worth the trouble which would be involved, in view of the probable substitution for the powder of fused metal or metallic alloys for steel-making purposes.

The blue oxide (Mo_3O_8) or its hydrate is soluble in water, so that merely shaking the sample with water serves to identify it; it is frequently present in the moist black powders. For the rest we have obtained a useful figure by boiling 1 grm. of the sample for five to ten minutes with 100 c.c. of a bi-normal solution of caustic potash, filtering, and precipitating the Mo in the filtrate. The oxygen is then calculated on the supposition that the Mo dissolved out existed as MoO_3. Metallic Mo is practically insoluble in potash, and, according to Comey's Dictionary, so also are Mo_2O_3 and MoO_2; hence the figure obtained would always err on the negative side.

The treatment with KHO occasionally results in the formation of a precipitate of lead sulphide in the filtrate when lead acetate is added for the estimation of the Mo. In such a case one must of course expel the H_2S from the acidified filtrate before precipitating.

Both Mo and MoO_2 reduce ammoniacal silver solutions, six atoms of silver being, according to E. F. Smith (*Journ. Chem. Soc.*, lxiv., p. 171), replaced by one of Mo. As silver can be estimated so easily and accurately the indirect estimation of the oxygen of a Mo powder by these means appeared promising. Unfortunately, however, with such highly carburetted (and sometimes sulphuretted) material as these powders, the results are always too high. One can of course distinguish a sample poor in metallic Mo from a rich one, but so far as a single sample is concerned the result forms only a rough test.

The following are typical analyses of commercial molybdenum alloys. I. and II. are molybdenum powders; III. and IV., MoNi alloys; V., a ferro-molybdenum; and VI., fused molybdenum metal.

	I.	II.	III.	IV.	V.	VI.
Mo	72·62	86·72	70·53	36·75	16·69	92·40
W	3·17	nil	1·27	—	1·53	0 74
Ni..	—	—	21·60	57·80	—	—
Fe..	1·60	0·48	3 08	1·60	76·34	2·73
C	7·80	8·18	2·94	1·08	4·20	3·87
Si	—	—	0·42	0·14	0·39	0·05
SiO_2	—	1·57	—	—	—	—
Mn	—	nil	0·40	trace	0·37	nil
S	—	0·10	trace	3·00	0·076	0·33
O	8·30	1·00	—	—	—	—
H_2O	4·30	—	—	—	—	—

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ELECTROLYTIC DETERMINATIONS AND SEPARATIONS.*

By LILY G. KOLLOCK.

(Concluded from p. 246).

Bismuth.

Smith and Thomas (*Am. Chem. Journ.*, v., 114) deposited bismuth from a citrate solution which contained an excess of sodium citrate. Bismuth hydroxide was dissolved in citric acid and sodium hydroxide added to alkaline reaction. Smith ("Electro-chemical Analysis," p. 69) also states that bismuth may be precipitated from an alkaline citrate solution and a solution containing free citric acid. The conditions under which the deposition took place are not recorded. The following experiments were accordingly made in order to obtain the proper working conditions:—

Bismuth from an Ammonium Citrate Solution.—A bismuth nitrate solution was made, adding sufficient nitric acid to the salt to prevent the formation of a basic nitrate on the addition of water. To a solution of this salt containing 0·1882 grm. of metallic bismuth were added 3 grms. of citric acid, and the solution made ammoniacal. It was diluted to 125 c.c. The temperature was raised to 70° C. A current of $N.D._{100}=0·02$ A and $V=1·8$ was allowed to act for three hours. At the expiration of this time the current was increased to $N.D._{100}=0·04$ A and $V=2·3$. Finally, in order to deposit the last traces of the metal held in solution, the current was made $N.D._{100}=0·09$ A. The deposit of bismuth obtained in this way was light grey in colour, very compact, and adherent. In one case the current acted all night, and the deposit was darker in colour, but adherent and easily washed. There was a noticeable amount of the bismuth peroxide deposited. The anode was weighed therefore before and after the electrolysis, and from the weight of the bismuth peroxide found, the amount of the bismuth deposited in this way, calculated and added to the weight of that precipitated upon the cathode (see Table XXIV.).

Bismuth from a Potassium Citrate Solution.—The solution of bismuth nitrate used contained 0·1777 grm. To this were added 7 grms. of citric acid, followed by an excess of potassium hydroxide. The solution was diluted to 125 c.c. The temperature was 70°. The current for this solution was also low at the beginning and was gradually increased to 0·09 A. The deposit of bismuth peroxide on the anode was treated as indicated in the preceding method. The bismuth was in all cases a grey compact adherent deposit (see Table XXV.).

Bismuth from a Citric Acid Solution.—Seven grms. of citric acid were added to a solution of bismuth nitrate containing 0·1767 grm. of bismuth. The solution at 70° was electrolysed with a current $N.D._{100}=0·02$ A and $V=1·8$. Subsequently it was increased to $N.D._{100}=0·07$ A and $V=2·6$ (see Table XXVI.).

Mercury from Nitric Acid Solution.—The fact that mercury could be determined satisfactorily from a nitric acid solution appears in the *Berichte* (xix., 329). Mercuric nitrate was electrolysed by Smith and Knerr (*Am. Chem. Journ.*, viii., 206). There was ½ to 1 c.c. of free nitric acid present. Bright shining deposits of 0·0911 grm. were obtained in thirty minutes. The current used for the purpose liberated 4 c.c. of oxyhydrogen gas per minute.

In the following work 3 c.c. of concentrated nitric acid were added to a solution of mercuric chloride containing 0·1403 grm. of metallic mercury. The solution (125 c.c.) was electrolysed with a current $N.D._{100}=0·06$ A and $V=2$ for four hours. The temperature was 70° C. The solution in the dish was syphoned off before the interruption of the current (see Table XXVII.).

Mercury from Sulphuric Acid Solution.—It has been known for a considerable time that mercury could be

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxi., No. 10.

TABLE XXIV.

Bismuth. Grm.	Citric acid. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Amount found. Grm.
0·1882	3	125	$N.D._{100}=0·02$ (for three hours.)	1·8	70°	16	0·1881
			$N.D._{100}=0·04$ A.	2·3	All night in cold.		
			„ =0·06 A.				
0·1882	3	125	„ =0·02 A. (for three hours).	1·6	70°	6½	0·1880
			$N.D._{100}=0·04$ A.	2			
			„ =0·09 A.	3			
0·1882	3	125	„ =0·03 A.	2	65°	6	0·1819
			„ =0·042 A.	2·58			
			„ =0·09 A.	3			
0·1882	3	125	„ =0·03 A.	2	65°	6	0·1820
			„ =0·042 A.	2·6			
			„ =0·09 A.	3			

TABLE XXV.

Bismuth. Grm.	Citric acid. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Amount found. Grm.
0·1767	7	125	$N.D._{100}=0·02$ A.	2·16	65°	5	0·17648
0·1767	7	125	„ =0·038 A.	2·18			
0·1777	7	125	„ =0·02 A.	2·5	70°	5	0·1781
0·1777	7	125	„ =0·05 A.	3			
0·1777	7	125	„ =0·03 A.	2·1	65°	5½	0·1774
0·1777	7	125	„ =0·05 A.	2·5			

TABLE XXVI.

Bismuth. Grm.	Citric acid. Grms.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Amount found. Grm.
0·1767	7	125	$N.D._{100}=0·02$ A.	1·8	65°	5	0·17648
0·1767	5	125	„ =0·38 A.	2·6			
			„ =0·02 A.	1·8			
			„ =0·05 A.	2·66	70°	5	0·1761

TABLE XXVII.

Mercury. Grm.	Nitric acid. C.c.	Dilution. C.c.	Current.	Voltage.	Temperature.	Time. Hours.	Amount found. Grm.
0·1403	3	125	$N.D._{100}=0·06$ A.	2	70°	4½	0·1404
0·1403	3	125	„ =0·07 A.	1·91	70°	4	0·1406

TABLE XXVIII.

Iron. Grm.	Citrate. Grms.	Citric acid. Grm.	Current.	Voltage.	Temperature.	Time. Hours.	Found. Grm.	Carbon. Per cent.
0·0378	10 c.c. = 1·18	3 c.c. = 0·059	$N.D._{100}=0·58$ A.	5·7	35°	7	0·0379	0·079
0·0378	10 c.c. = 1·18	3 c.c. = 0·059	$N.D._{175}=8$—1·3	9—11	70°	4	0·0383	1·1
0·1035	10 c.c. = 1·18	3 c.c. = 0·059	$N.D._{175}=8$	9—11	50°	4½	0·1046	1·06
0·1277	10 c.c. = 1·18	5 c.c.	$N.D._{100}=0·4$—1 A.	7—11	50°	4½	0·1278	0·00
0·1277	10 c.c. = 1·18	5 c.c.	„ =0·8 A.	7—8	50°	4½	0·1280	0·94

deposited from a solution feebly acidulated with sulphuric acid. The current which was used generated 5 to 6 c.c. of oxyhydrogen gas per minute. In repeating this method for the precipitation of mercury, 1 c.c. of sulphuric acid was added to a solution of mercuric chloride. A current of $N.D._{100}=0·4$—0·6 A and V=3·5 effected the deposition. The temperature during the operation was 65° C. The mercury was entirely deposited in one hour; it had a brilliant metallic appearance; it was washed with cold water. The mercury present in the solution was 0·1403 grm. The deposit weighed 0·1402 grm.

Iron from a Citrate Solution.—In 1888 Smith reported the deposition of iron from a sodium citrate solution containing free citric acid. The solution used was ferric ammonium sulphate, and the current acting upon the solution gave 6 c.c. of oxyhydrogen gas per minute.

The method was repeated, using a solution of the alum containing 0·0378 grm. of metallic iron, 10 c.c. of sodium citrate (equivalent to 1·18 grms.), and 3 c.c. of citric acid (equivalent to 0·059 grm.), and the solution diluted to 125 c.c. With a current of $N.D._{100}=0·58$ A and V=5·7 in seven hours the deposition was complete. Another determination gave 0·0375 grm. of iron, and a third gave 0·0379 grm. By titration with potassium permanganate the deposit was found to contain 0·03789 grm. of iron. The carbon therefore in this deposit was 0·079 per cent.

Similar experiments were conducted, using 0·1035 grm. of iron. The current used was N.D.$_{175}$=0·8 A and V=9 The temperature was 60° during the electrolysis. The solution was 250 c.c. The deposit weighed 0·1046 grm. By titration of the iron, 1·06 per cent of carbon was found. With identical conditions, using 0·1280 grm. of iron, 0·94 per cent of carbon was found deposited in the metal (see Table XXVIII.).

NOTE ON THE ESTIMATION OF NICOTINE, AND ON THE AMOUNT OF NICOTINE IN NEW SOUTH WALES TOBACCOS.*

By G. HARKER, B.Sc.

Kissling's and Biel's methods for nicotine were compared and found to give similar results. The generally accepted statement that nicotine is lost from its ethereal solution during evaporation was not found in accord with the results obtained in this investigation. An alternative method given by Biel, in which the weight of the double sulphates of nicotine and ammonia was employed, was found to be practically worthless, as a constant weight cannot be obtained. Several attempts, all of which occupied much time, were made to estimate nicotine volumetrically in an aqueous solution containing nicotine and ammonia, but owing to the close similarity in the properties of these substances the attempts were not successful. Four varieties of New South Wales tobaccos were analysed for nicotine, with the following result:—

Manilla	1·95 per cent
Tamworth	2·36 ,,
Tumut	3·84 ,,
Bathurst	4·53 ,,

THE RELATION OF THE TASTE OF ACID SALTS TO THEIR DEGREE OF DISSOCIATION.

By LOUIS KAHLENBERG.

Investigations on the taste of dilute solutions of acids, when regarded in the light of the theory of electrolytic dissociation, have yielded the general result that sour taste is due to hydrogen ions, and that its intensity varies as the concentration of the hydrogen ions in the solution. According to Richards (*Amer. Chem. Journ.*, xx., 121), sulphuric, nitric, hydrobromic, and hydrochloric acids can still be detected by the sense of taste in solutions somewhat less than *n*/1000. My own tests place the limit at *n*/800, at which dilution the taste is only astringent, while at *n*/400 it is slightly sour. It will be seen from this that the sense of taste is indeed a fairly delicate means of detecting acidity. Richards observed that while the sour taste of tartaric, citric, and acetic acids diminishes in the order named, and hence agrees qualitatively with the dissociation theory, the quantitative agreement is not satisfactory, acetic acid, for instance, having a sour taste at least as strong as hydrochloric acid of one-third the equivalent concentration, whereas the former acid is dissociated to only about one fourteenth the extent of the latter. The results of my tests (L. Kahlenberg, *Bull. Univ. of Wis.*, ii., 1—31; ref. *Journ. Phys. Chem.*, 1899, iii, 66; *Zeit. Phys. Chem.*, 1899, xxix., 343), with acetic acid are similar to those of Richards, for I found that acetic acid has about four times as sour a taste as one would expect according to the dissociation theory.

The object of this article is to investigate the taste of the acid sodium salts of a number of dibasic organic acids, and also of the acid salts of citric acid, and to view the results in the light of the theory of electrolytic dissociation.

The acid sodium salts of the following acids were tested: Oxalic, malonic, succinic, malic, tartaric, fumaric, maleic, and citric. The method of investigation was as follows: Solutions containing 1 grm.-molecule of the acid salts in 100 litres were prepared; these were diluted successively with equal volumes of water. All of the solutions were tasted by myself by holding 6 to 8 c.c. in the mouth for a few months after it had been well rinsed with distilled water. Of course the results can not claim to be as free from bias as those that were obtained in my former investigations, where a number of persons who were entirely ignorant of the nature of the substances they were tasting, tasted the solutions. My previous work, however, has taught me that my own sense of taste is what one would call normal; and the data given below I deem perfectly reliable, as I did not work with solutions so dilute that their taste was not clearly defined. The taste of solutions of the acid sodium salts of malic and tartaric acids was compared with that of a very dilute solution of hydrochloric acid of known strength. In these tests the method of experimentation employed in my previous investigations (*loc. cit.*), was strictly followed, five persons serving as subjects. The results of these tests are also given below.

The degree of the hydrogen dissociation of acid sodium salts has been determined by means of the method of sugar inversion by J. E. Trevor (*Zeit. Phys. Chem.*, 1892, x., 321), and also more recently by W. A. Smith (*Zeit. Phys. Chem.*, 1898, xxv., 217). The figures found are, as is well known, surprisingly low. The following table gives the percentage of hydrogen dissociation in the case of the acid sodium salts in question, and also the volume in litres in which 1 grm. of hydrogen ions is contained. With the exception of the figure for oxalic acid, the results are those of Smith. With but few exceptions the results of Trevor and those of Smith agree tolerably well. For the present purpose it is immaterial whether the results of the one or those of the other be used, as the general conclusion would remain the same.

Acid sodium salt of—	H dissociation in per cent when 1 grm.-mol. is present in 128 litres.	1 grm. H ions is contained in— Litres.
Oxalic acid	4·37	2929
Malonic ,, ...	0·50	25600
Succinic ,, ...	0·159	80500
Malic ,, ...	0·719	17800
Tartaric ,, ...	2·800	4571
Fumaric ,, ...	1·52	8421
Maleic ,, ...	0·55	23273
Citric ,, ...	—	—
Mono Na salt	1·92	6666
Di Na salt	0·060	213333

Assuming that sour taste is due to hydrogen ions, it follows from these results (and the fact above mentioned, that the sense of taste is able to detect the presence of the strong mineral acids at no greater dilution than 1 grm.-molecule in 1000 litres) that none of the salts given in the table ought to have any sour taste at all when 1 grm.-molecule is contained in 128 litres. Now as a matter of fact they all have a sour taste at this dilution, and some of them even at much higher dilutions, as the following data show.

Acid sodium oxalate is very plainly sour at the concentration 1 grm.-mol. in 200 litres, and even at 1 grm.-mol. in 400 litres it is still distinctly, though of course faintly, sour.

Acid sodium malonate is distinctly, though slightly, sour at 1 grm.-mol. in 200 litres; while at 1 grm.-mol. in 400 litres it is slightly astringent.

A solution of acid sodium succinate containing 1 grm.-mol. in 200 litres is still distinctly sour, but it also has the disagreeable taste characteristic of the free acid.

Acid sodium malate is distinctly sour at 1 grm.-mol. in 200 litres, but slightly astringent at 1 grm.-mol. in 400 litres.

* Abstract of a Paper read at the Melbourne Meeting of the Australasian Association for the Advancement of Science, Jan., 1900.

A sodium bitartrate solution containing 1 grm.-mol. in 200 litres is very distinctly sour; while at 1 grm.-mol. in 400 litres it is astringent or very slightly sour.

Acid sodium fumarate is distinctly sour at 1 grm.-mol. in 100 litres, and still plainly, though faintly, sour at one-half this strength. At 1 grm.-mol. in 400 litres is slightly astringent.

The acid sodium salt of maleic acid tastes sour at 1 grm.-mol. in 100 litres, and it has in addition the disagreeable taste characteristic of maleic acid. At 1 grm.-mol. in 200 litres the acid salt has a slightly astringent effect.

Mono-sodium citrate is very plainly sour at 1 grm.-mol. in 200 litres; while at 1 grm.-mol. in 400 litres it is weakly but still distinctly sour. At one-half the latter strength the solution is very faintly sour or astringent.

Di-sodium citrate is slightly sour at 1 grm.-mol. in 100 litres; while at one-half this concentration it is very faintly sour or simply astringent.

In addition to this, I found by direct tests* on five persons that a solution of acid sodium malate of 1 grm.-mol. in 50 litres is about as sour as a solution of hydrochloric acid containing 1 grm.-mol. in 140 litres; again, a solution of acid sodium malate of 1 grm.-mol. in 100 litres is about as sour as a solution of hydrochloric acid of 1 grm.-mol. in 320 litres. A solution of sodium bitartrate containing 1 grm.-mol. in 100 litres is as sour as a solution of hydrochloric acid containing 1 grm.-mol. in from 280 to 300 litres. A difference in the quality of the tastes of these dilute solutions of hydrochloric acid and the sodium bimalate and sodium bitartrate respectively could not be detected by the persons tested or by myself.

It is clear from the facts presented that the sour taste of solutions of these acid sodium salts can not be ascribed to the hydrogen ions present, for there are too few of them. The question then arises—to what is the sour taste of these solutions due? Looking at the matter from the standpoint of the theory of electrolytic dissociation, the undissociated molecules and also the bivalent anions in the solutions in question are relatively very few in number, while sodium ions and monovalent anions of the general formula $\overline{H}Ac$ are abundant. Now as sour taste is not produced by sodium ions (for solutions of the normal sodium salts have no sour taste), it follows that it must be ascribed to the monovalent anions. We are led to the conclusion, then, that these particular anions produce upon the sense of taste an effect that is identical with that produced by hydrogen ions. It should be borne in mind in this connection that these anions are large complexes varying in chemical composition in the case of different salts, that they bear electrical charges opposite to those on hydrogen ions, and that they are much less mobile than the latter. It is therefore evident that the explanation of the sour taste of the solutions of these acid salts from the standpoint of the theory of electrolytic dissociation is unsatisfactory to say the least.—*Journal of Physical Chemistry*, iv. No. 1.

CORRESPONDENCE.

SYNTHESIS OF ARSENIC.

To the Editor of the Chemical News.

SIR,—On page 257, col. 2, lines 9 and 10 of the current volume of the CHEMICAL NEWS, I read:—"[*Note by Translator.* — There is evidently an error in these figures]." Allow me to point out that the figures given, viz., the percentage of arsenic yielded by certain weights of phosphorus, are perfectly correct.—I am, &c.,

ANALYST.

London, W., June 2, 1900.

* These experiments were conducted in the same way that my previous tests on taste were made (*l. c.*), so that a detailed description is hardly necessary in this connection.

NOTICES OF BOOKS.

Geber; A Tale of the Reign of Harun al Raschid, Khalif of Baghdad. By KATE A. BENTON. New York: Frederick A. Stokes Company. 1900. Pp. 487. 8vo.

THE name Geber brings to mind those noted chemical treatises that for four centuries have been attributed to the famous Arabian physician, but which Berthelot has shown to be fraudulent. In the romance before us Mrs. Benton has taken the astrologer and physician as a central figure, and has grouped around him historical personages and imaginary characters in a sumptuous oriental setting.

The characters of the famous Khalif and of Jaafer the Barmecide and his family closely follow the lines of history, but the events which led to the rupture of the friendship between Harun and Jaafer, and the consequent downfall of the latter's family, while mainly historically correct, contain much that is fiction.

The story opens with the call of the physician Geber to Damascus, where the favourite wife of the Khalif is suffering from a mysterious malady; the astrologer, who has his own reasons for enmity against Harun, sees in the call an opportunity to work a long meditated revenge, obeys the summons, and with his family and servants takes up his abode in the magnificent capital of the Khalif's dominions, earning his gratitude and many privileges by effecting an immediate and remarkable cure.

The wily astrologer makes good use of his advantages, and slowly weaves a web around Harun wounding him in all his most vulnerable points, and finally making him believe that his dearly beloved wife is unfaithful, and his trusted friend is his betrayer.

The interest is well sustained, although the great numbers of characters introduced is sometimes confusing to the reader. Several love stories run their course through the book, two of which, after due allowance of danger and difficulty, end in the orthodox happy manner.

Of genuine alchemy, either theoretical or practical, the book contains little, the romantic side of the period being that which is more fully developed by the gifted author; there are, however, several scenes in which astrology and catoptromancy play their part.

Perhaps the weakest incident in the book, because most improbable and at variance with what the writer has shown to be the true character of the leading figure, is the closing scene, where Geber having accomplished his revenge and seen his desire upon his enemies, sparing none who stood in the way of his purpose, becomes a Christian and dies with words of repentance and charity upon his lips.

The book has a pathetic interest, the author having died while it was going through the press. There is a bibliography of authors and the volume shows the result of much painstaking study. Some reviewers have pronounced the book to be one of the most fascinating stories published during the year.

Flame, Electricity, and the "Camera." Man's Progress from the First Kindling of Fire to the Wireless Telegraph and the Photography of Colour. By GEORGE ILES. New York: Doubleday and McClure Co. 1900. Pp. xv.—398, 8vo. Ill.

To trace man's progress in applied science from the first kindling of fire to the latest discoveries made in the year 1899 is a vast undertaking, but Mr. George Iles is to be congratulated on the manner in which he has accomplished the task: he has produced a book as fascinating to the student of science and of civilisation as a clever romance is to a reader of good fiction, and he has been fortunate in finding a publisher who knows that a strong book deserves a handsome dress.

Mr. Iles is a pronounced evolutionist, and writes from that standpoint; the main argument of the book was indicated by the author in the *Popular Science Monthly* for June, 1876. He claims that progress in scientific discovery obeys a law of permutative multiplication, that here and there the progress of humanity has made *leaps*, and three of the most prodigious bounds were made when heat, electricity, and the camera became the servitors of the race. In pourtraying this conception he is fortunate in possessing a charming and forcible diction, while he rarely neglects a small point which can strengthen his argument. Indeed very little seems to have escaped close study; he is equally at home in describing the mastery of metals, the conversion of heat into motive power, the methods of producing electricity from the first beginnings to the latest improved apparatus; special instruments, such as the telephone, the kinetoscope, and the bolometer, as in depicting the forward march of the sciences underlying these, and their application to everyday life.

In a book covering so enormous a range, condensation is of course a prime necessity, but Mr. Iles is never obscure: he has the faculty of generalising as well as of specialising, and, whether he is describing the problems of multiplex telegraphy, or those of colour-photography, the discovery of Röntgen's X-ray, or the history of Cyrus Field's courage in laying the Atlantic cable, he never fails to interest the reader.

The author means to be impartial, and if he brings into prominence the work of Americans in the diverse fields, can he be blamed? Especially as they are sometimes neglected by Europeans writing on the history of science. He has been a careful student of the chronology of facts, but the reviewer believes that on page 271 he gives credit to Porta for discoveries made long previously by the wonderful man Leonardo da Vinci.

The volume is not a history, yet it contains much history; it is not a text-book in any sense, yet it could be most advantageously read by students of science; it is not a romance, yet it treats in most agreeable language of the romantic side of science.

It is interesting to note how very recent in date are the occasional citations; one gives statistics as late as December, 1899. This is only an indication of the up-to-date-ness of the whole work. Ninety-three illustrations and twenty-two superior plates illustrate the volume; the portraits of Volta, Faraday, Lord Kelvin, Edison, Niepce, Daguerre, and the artistic plates, are of the best.

No one who cares to inform himself as to the steps by which modern civilisation has reached its present plane can afford not to *own* this remarkable book.

H. C. B.

Practical Methods for Determining Molecular Weights. By Henry Biltz. Translated by H. C. Jones and S. H. King. Easton, Penna.: The Chemical Publishing Company.

The subject of this work is, as its title implies, a description of the theory and practice of the various methods devised for the determination of molecular weights. Some of these have been well known for many years, whilst others have been more recently devised, and are still buried in technical periodicals and pamphlets.

The first method treated, that of vapour density, depends on Avogadro's law, that, at a given pressure and temperature, an equal number of molecules of all gases occupy the same volume. This was early developed, on two different lines, by Gay-Lussac and Dumas; both being limited in their range of application, were superseded by V. Meyer's method of gas displacement. A useful list of liquids suitable for employment in the vapour-jacket, at various temperatures, appears on page 18. Hofmann's method of mercury displacement is also described, though it is seldom used nowadays.

The second method described is the Osmotic. This has been principally applied in three different manners, viz., (1) the freezing-point method; (2) the boiling-point method; (3) Nernst's method of observing the lowering of solubility. These are treated at great length, and their uses and limitations fully set forth; the principal objection is that the molecular weight cannot be directly determined by these means, but must be arrived at by extrapolation on the curve of plotted results.

Traube's method, discovered and worked out in the years 1895-6, requires a pre-knowledge of the composition of the substance under investigation and an approximate knowledge of its formula. There is at present no theoretical foundation for this method; the principle upon which it depends is this:—"An increase in volume takes place when a molecule is formed from atoms; this is independent of the chemical nature of the substance, and can be only slightly modified by constitution. It amounts to 25·9 c.c. for a grm.-molecule at 15° C."

The book is worthy of a place in all laboratories where general analytical work is being carried on.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et de Chimie, Series 6, Vol. xi., No. 5.

Analysis of Water from Jouhe, near Dôle (Jura).—Paul Bourcet.—The waters of Jouhe come from a source of which the therapeutic value has been widely spread in the neighbourhood. Though famous so long ago as the year 1710, it appears that nothing was known definitely as to the composition and saline richness of these waters; the author has therefore made an analysis. The sample was taken on August 17, 1899; the temperature of the water was +12·7°, the temperature of the air being +22·7°; its appearance was clear, and it had a slight sulphurous smell. One litre of water gave off, when boiled, 29·2 c.c. of gas at 760 m.m. B.P. and 15° C. The composition of this gas was found to be:—

	Per cent.
Carbonic acid	29·35
Oxygen	7·32
Nitrogen	61·16
Marsh gas	2·17

and a trace of sulphuretted hydrogen. After evaporation, this water left a residue weighing 2·8347 grms., of the following composition:—

	Grms.
Chloride of sodium	1·3203
Chloride of magnesium	0·2596
Bicarbonate of magnesia	0·0105
Sulphate of lime	0·9973
Bicarbonate of lime	0·2498
Carbonate of iron	0·0004
Silica	0·0097
Iodine	traces
	2·8476

In spite of the local opinion, this water is not sulphurous. The faint odour of sulphuretted hydrogen which it possesses comes from a slight decomposition of the sulphate of lime by the organic matter present.

Composition of Essence of Santal from the East Indies.—M. Guerbet.—The result of a large number of experiments on the composition of essence of santal may be represented approximately in the following manner:—

Santalenes α and β	60
Santalols α and β	800
Santalal	30
Acids in the form of ethers (formic, acetic, santalic, teresantalic)	30
Undetermined products, with a powerful odour, boiling at 130—220°	3
Undetermined products, boiling at about 320° and over (carbides, alcohols, ether oxides, resinous product)	77
	1000

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 2.

The Action of Mercury on Iodide of Methylene.—V. Thomas.—Already inserted.

Preparation of Carbazides. Action of Hydrazines on the Phenolic Carbonates.—P. Cazeneuve and M. Moreau.—When 25 grms. of hydrazine in solution in 25 grms. of water are poured into 25 grms. of carbonate of phenyl, the mixture becomes very hot and the carbonate dissolves. On cooling, a magnificent crop of crystals of phenate of hydrazine are obtained, which melt at 63—64°, and by analysis correspond with a compound of four molecules of phenol to one molecule of hydrazine, $(C_6H_6O)_2,NH_2—NH_2(C_6H_6O)_2$. This phenate is easily decomposed. When one molecule of phenylic carbonate, or 25 grms., is heated for an hour to 160—170° with four molecules of phenylhydrazine, or 50 grms., traces of CO_2 are given off, and the liquid gradually becomes yellow. On cooling, a yellowish white crystalline mass is obtained, which is taken up warm with 200 grms. of alcohol at 60° made strongly acid with hydrochloric acid to remove the excess of phenylhydrazine. After two re-crystallisations, a body is obtained, melting at 169—170°, corresponding to $CO<^{NH—NH—C_6H_5}_{NH—NH—C_6H_5}$. In a similar manner two other bodies are obtained by heating together methyldiphenylhydrazine with carbonate of phenyl and naphthylhydrazine β with phenylic carbonate. This first one, having the formula $CO<^{NH—NCH_3—C_6H_5}_{NH—NCH_3—C_6H_5}$, melts at 149—150°, and cannot be distilled without decomposition; the second body, $CO<^{NH—NH—C_{10}H_7\,\beta}_{NH—NH—C_{10}H_7\,\beta}$, does not melt without decomposition.

Transformation of the Acetic Ethers of Ordinary Phenol, Paracresol, and Thymol into Benzoic Ethers.—F. Bodroux.—Fifteen grms. of chloride of benzoyl were placed with 10 grms. of acetate of phenyl in a flask of 200 c.c. capacity. A few grms. of powdered zinc were added to the mixture; at first nothing happened, but after a time the mixture warmed up and a violent reaction took place, giving rise to an abundant disengagement of hydrochloric acid. This soon stops, and the product, when left to itself, becomes solid in a few hours. This solid body, after drying, is dissolved in equal parts of warm alcohol and water, and on cooling forms large colourless prisms melting without decomposition at 69°; this body has been identified as benzoate of phenyl. This manner of acting in the presence of zinc powder is not peculiar to acetate of phenyl only; the same reaction takes place with the acetic ethers of paracresol and thymol, and the author has obtained the benzoïc ethers of these two phenols. On the other hand, with acetate of naphthol β there is no reaction produced except towards 80°, but this is due simply to the decomposition of the chloride of benzoyl by the zinc, and at the end of the operation the acetic ether can be recovered unchanged.

Naphthopurpurine, a Product of the Oxidation of Naphthazarine.—G. F. Jaubert.—Already noticed.

Volumetric Estimation of the Benzenic Quinones.—A. Valeur.—Already noticed

MISCELLANEOUS.

University of London.—Rogers Prize.—At a meeting of the Senate of the University of London, held on March 28th, 1900, it was resolved that one sum of £100 be offered as the Rogers Prize open for competition to all the members of the medical profession in Great Britain and Ireland, for an essay under the following regulation as to the subject and conditions thereof, namely:—An essay by any member of the Medical Profession in Great Britain and Ireland upon the "Production of Immunity in Specific Infective Diseases"; generally, and with particular reference to any one disease on which the writer of the essay has made original investigations. The essay is to be sent to the Registrar, University of London, South Kensington, S.W., on or before June 1st, 1901.

The Synthesis of Chrysine.—MM. Emilewicz, Kostanecki, and Tambor.—The authors effected the synthesis of chrysine in the following manner:—They reacted with the ethylic ether of benzoic acid on the trimethylic ether of phloracetophenone in the presence of metallic sodium; a δ-diketone is formed, this and the 2.4.6-trimethoxybenzoylacetophenone under the influence of hydriodic acid are demethylated, and by the closing of the chain is transformed into chrysine. The chrysine thus obtained (1·3-dioxyflavone), $C_{15}H_{10}O_4$, has exactly the properties of the chrysine described by Piccard, but to characterise it and identify it still better with this compound, the authors have prepared tectochrysine with their product (1-oxy-3-methoxyflavone), $C_{16}H_{12}O_4$, which has the fusion point expected, viz., 163°, and crystallises in benzene, as shown by Piccard, in large sulphur-yellow crystals. These crystals will be measured. The authors propose to generalise the reaction which has led them to this interesting synthesis, and they rightly point out the value of the services rendered by Piccard to this subject, when he examined and described the colouring matters in poplar buds.—*Berichte*, xxxii., p. 2448.

MEETINGS FOR THE WEEK.

MONDAY, 11th.—Society of Chemical Industry, 8. "The Maintenance of Aëration as a Standard of Purity of Sewage Effluents," by W. J. Dibdin, F.I.C., F.C.S., and G. Thudichum, F.C.S. "The Composition and Determination of Cerium Oxalate," by Frederick B. Power, Ph.D., and Frank Shedden, B.Sc. "The Production of Nitrate of Soda in Chili," by F. G. Welch.

— Royal Institution, 5. General Monthly Meeting.

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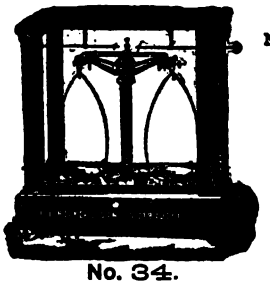

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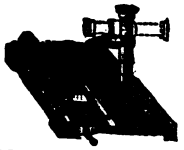

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VAPOUR-DENSITY OF BROMINE AT HIGH TEMPERATURES.*

(Supplementary Note).

By E. P. PERMAN, D.Sc., and G. A. S. ATKINSON, B.Sc.

The authors regret that they had overlooked a monograph by C. Langer and V. Meyer, entitled "Pyrochemische Untersuchungen," containing an account of some experiments on the vapour-density of bromine. Their method was to pass a mixture of bromine vapour and nitrogen into a porcelain tube with capillary ends placed in a furnace in a horizontal position. The bromine and nitrogen were then displaced by a current of carbon dioxide, the bromine being absorbed by potassium iodide solution, and the carbon dioxide by potash solution, while the nitrogen was collected and measured. Temperature was ascertained by displacing the tube full of air in a similar way. By this method Langer and Meyer carried out experiments at "Zimmer-temperatur," 100°, 900°, and 1200°.

The only results comparable with the authors' are those at 900°. Their results at this temperature are 5·478, 5·414, 5·433, 5·382, 5·59; mean, 5·459 (air = 1), or 78·88 (H = 1). They say that *these results indicate that the vapour-density of bromine, even when diluted with eleven times its volume of air, is still normal at* 900°.

The mean of our results at this temperature is 78·6, and the density read from the curve (*Roy. Soc. Proc.*, vol. lxvi., p. 17) is 78·8. This close agreement shows that Langer and Meyer's results really indicate a small amount of dissociation at 900°. Diminution of pressure appears to have little effect at that temperature, as Langer and Meyer used bromine much diluted with nitrogen, while in our experiments there was no decrease of density on reducing the pressure from 767 m.m. to 365 m.m.

It may be noted that Langer and Meyer give the boiling-point of bromine as 63°, whereas we have found the boiling-point of specially purified samples to be very close to 58·9°. (See Ramsay and Young, *Trans. Chem. Soc.*, vol. xlix., p. 454, *et seq.*).

TESTING MINERALS WITH ORGANIC ACIDS.

A Claim for Priority.

By H. CARRINGTON BOLTON, Ph.D.

The *Journal of the American Chemical Society* for March, 1900, contains a paper by Joseph W. Richards and Norman S. Powell, entitled, "Substitutes for Hydrochloric Acid in Testing Carbonates"; the authors find that potassium acid sulphate, oxalic acid, citric acid, and tartaric acid can be used in testing carbonates, producing effervescence more or less actively, and they give a table of results. The authors make no reference to previous work on the same lines, and this prompts me to make the claim that about twenty years ago I anticipated all their observations, and published the results in periodicals accessible to every one.

Between 1877 and 1882 I published three memoirs under the title, "Application of Organic Acids to the Examination of Minerals," in which I showed the action of these acids on 200 mineral species, including carbonates, sulphides, oxides, silicates, and many others. I pointed out the usefulness of citric acid as a substitute for hydrochloric acid in the laboratory and in the field, and showed that by means of it certain minerals could be readily distinguished.

These papers were printed in whole or in part in the following journals :—Chem. News, vols. xxxv., xxxvi., xliii., and xlvii.; *Annals N. Y. Acad. Sciences*, vols. i. and ii.; *Proceedings Amer. Assoc. Adv. Science*, vol. xlvii.; *Reports Brit. Assoc. Adv. Sci.*, vol. l.; *Mineralogical Magazine*, vol. iv.; *Berichte d. D. Chem. Gesellschaft*, vol xiii.; and abstracts appeared in many other journals. Moreover, the use of citric acid in testing minerals was adopted by teachers in more than one scientific school; the use of potassium acid sulphate having been known long before. Finally, Nason's edition of Elderhorst's "Manual of Qualitative Blowpipe Analysis" (1881) gives a whole chapter to my methods.

It is gratifying to note that the results obtained by Richards and Powell agree well with mine. Coincidences of independent thought often occur, but in publishing a research some acknowledgment of a previous work is generally made; had the authors made even a slight examination of familiar literature they would have found that their field of study had been thoroughly traversed.—*Journal Am. Chem. Soc.*, May, 1900.

Washington, D.C., March 20, 1900.

PRODUCTION OF OZONE BY THE DECOMPOSITION OF WATER BY MEANS OF FLUORINE.

By HENRI MOISSAN.

When during any reaction the oxygen is set free at a low temperature, we observe that this simple body becomes polymerised with the greatest ease, and that ozone is formed. As an example we may mention the action of sulphuric acid on binoxide of barium or on permanganate of potash. It is true that if the reaction causes any production of heat the ozone is destroyed and we only find traces of it; by reason of the instability of ozone at the ordinary temperature its destruction may even be complete.

The action of fluorine on water furnishes a new proof of this easy polymerisation of oxygen at a low temperature.

We showed in 1891 that fluorine in the presence of water decomposes this liquid at the ordinary temperature, with the formation of hydrofluoric acid and ozone. We even remarked that by allowing a few drops of water to fall through an atmosphere of fluorine, the ozone produced was sufficiently concentrated to appear with the beautiful blue colour observed by MM. Hautefeuille and Chappuis.

We have repeated these experiments with the aid of a more abundant current of fluorine prepared in a copper vessel, and we were thus enabled to pass a large volume of fluorine through a small quantity of water.

The fluorine is passed by a small platinum tube through a water jacket kept at a constant temperature of 0°. It then passes into a Chancel flask with a round bottom, similar to those used for taking the density of gases. When this flask is filled by displacement with ozonised oxygen, this latter is titrated by means of a solution of iodide of potassium, in presence of an excess of sulphuric acid to prevent the formation of iodate. The iodine set at liberty is finally estimated by hyposulphite of sodium.

To transfer the solution of iodine into the flask without losing any ozone, a long-stemmed funnel is fixed to the central tube, and the liquid, to which sulphuric acid is added, is poured in. The flask is then thoroughly cooled down by means of carbonic acid and acetone. The gas contracts, and on opening the tap the liquid runs into the interior

* A Paper read before the Royal Society, May 31, 1900.

The solution of iodide of potassium in sulphuric acid is in this manner run in several times until the gas no longer colours the iodide.

The end of the reaction is recognised by allowing the flask to take the temperature of the laboratory and then letting a bubble of the gas pass through the tap into the funnel, in which there remains a little iodide solution. This bubble ought not to produce any colouration. After shaking, the flask is uncorked and the free iodine estimated by means of hyposulphite.

The following experiments show the regularity with which they can be carried out :—

Length of experiment.	Ozone per litre. Volume.	Weight.
5 minutes	56·3 c.c.	0·1207 grm.
10 ,,	90·7 ,,	0·1945 ,,
30 ,,	143·9 ,,	0·3085 ,,

By reducing these figures to percentages we get :—

5 minutes	5·63 c.c.
10 ,,	9·07 ,,
30 ,,	14·39 ,,

According to this experiment the proportion of ozone produced in the gas was, by volume, 14·39 per cent. After this moment the quantity of ozone present remained practically constant. As the figures show, it is a fairly high proportion. In reality the concentration of the ozone produced by fluorine in contact with water is much greater than that shown by previous analyses. As a matter of fact the displacement of the air in the flask by the ozone requires a fairly long time, during which the concentrated ozone decomposes.

On the other hand, the influence of the speed of the current is very great. In our experiments the speed was 3 litres per hour. The more rapid the current of fluorine, taking care, of course, to keep the temperature down to 0°, the more concentrated will the ozone be. An equilibrium is here formed between the two following reactions :—

1. The formation of blue ozone by decomposition of the water by an excess of fluorine.
2. Destruction of the ozone by increase of the surrounding temperature.

In several series of experiments, when the speed of the current of fluorine was less than 2 litres an hour, the proportion of oxygen to ozone varied from 10 to 12 per cent. In other experiments in which the bulb containing the water was not kept cooled down to 0°, the proportion of ozone was much lower.

The easy formation of concentrated ozone by the action of fluorine on water at a temperature of 0°, may possibly become the starting point of several applications.

The preparation of fluorine by the electrolytic method is certainly a delicate operation, but it is not expensive. Further, the ozone obtained by this means does not contain a trace of oxygenated nitrogen compounds. In this method of preparing ozone all secondary reactions are prevented, and if this new method becomes in any way commercial, it will afford great evidence of the chemical activity of fluorine.—*Bull. Soc. Chim.*, Ser. 3, xxiii., No. 8.

New Work on Acetylene Gas.—Professor Vivian B. Lewes has in the press an exhaustive work on "Acetylene Gas, a Handbook for the Student and Manufacturer." The book contains over 250 illustrations, and comprises a history of acetylene, its preparation, properties, and chemical reactions, together with a complete list of legal enactments in full concerning its use and manufacture, patents, and other important data. Messrs. Archibald Constable and Co. are the publishers.

RECENT DEVELOPMENTS IN THE ACETYLENE INDUSTRY.

By M. LUNDSTRŒM.

As the result of a large number of analyses the author gives the following figures for the composition of acetylene now made from commercial carbide of calcium :—

	Minimum.	Maximum.
Sulphuretted hydrogen	0·0	1·34
Phosphoretted hydrogen.. ..	0·03	1·7
Siliciuretted hydrogen	0 0	0·8
Arseniuretted hydrogen	0·0	0·004
Ammonia	0·06	2·8
Carbonic oxide	0·0	1·48
Hydrogen	0 07	0·27
Nitrogen	0·2	2·91
Oxygen	0·55	1·18

In the event of insufficient cooling during the preparation, the acetylene may also contain benzene vapour.

In a general way commercial carbide of calcium gives a gas containing 99·5 per cent of acetylene and 0·5 per cent of other gases. The oxygen and the nitrogen come from the air enveloping the pieces of carbide. The presence of hydrogen is due to the metallic calcium formed by the dissociation of the carbide at a high temperature. The raw acetylene may even contain as much as 20 per cent of this gas.

The siliciuretted hydrogen probably comes from the silica which is always present in the lime used; as for the ammonia, it comes from the nitride of magnesium. Finally, the sulphuretted hydrogen is due to sulphide of alumina, and the phosphoretted hydrogen to phosphide of lime.

A large number of methods have been proposed for the purification of acetylene. Lunge and Cedercreutz recommend chloride of lime; Frank proposes acid solutions of metallic salts (ferric chloride or cupric chloride in hydrochloric solution); Ullmann uses an acid chromic solution; and Stern passes the gas through petroleum, alcohol, or benzene. In actual practice nearly all the impurities are absorbed by the lime-water, and a simple washing with water will even suffice, unless the gas contains a large quantity of phosphoretted hydrogen, in which case a more complete purification will be necessary. According to the author Frank's method should be preferable to any other.

Carbide of calcium and acetylene have found numerous applications besides lighting; for example, for the complete dehydration of alcohol. In this operation it is necessary to use anhydrous sulphate of copper to absorb the acetylene set free.

Acetylene can also be used for the preparation of extremely fine lamp-black. Sœderbaum, again, uses it for the quantitative separation of copper from zinc, cadmium, and arsenic.

Finally, Bunsen burners have been made by means of which acetylene can be used for heating purposes.—*Chem. Gesell. Stockholm*, 1899.

NEW PREPARATIONS OF SOME OXIDES OF URANIUM.

By J. ALOY.

Uranous Oxide or Crystallised Suboxide of Uranium, UO_2.—There are several methods of obtaining this compound:—1. Arfwedson prepared suboxide of uranium, which he took for a metal, by heating the double chloride of suboxide of uranium and potash in a current of hydrogen and removing the alkaline chloride by lævigation; 2. Wöhler obtained UO_2 in the form of a black crystalline powder, by evaporating the hydrochloric solution of uranate of ammonium and sea-salt to dryness and then

fusing; 3. A more convenient method, due to M. Ditte, consists of calcining the oxide U_3O_5 moistened with hydrofluoric acid in a platinum crucible; 4. Finally, the reduction of the oxides U_3O_8 and UO_3 by carbon, also leads to the formation of crystallised UO_2.

To these methods the following, which is remarkably simple, may be added: to obtain the crystallised oxide, UO_2, it suffices to heat crystallised uranic hydrate, the preparation of which is described further on, in a current of hydrogen. In this manner this hydrate is transformed into a brilliant black powder, composed of small crystals, which, examined under the microscope, appear in the form of rectangular orthorhombic plates, for the most part modified by the four angles being truncated. The uranous oxide, UO_2, thus produced is not pyrophoric; it is even but slightly oxidisable; it must be kept for a long time at a red heat to be transformed into the green oxide. Like the other varieties of oxide, amorphous or crystallised, it is insoluble in dilute hydrochloric and sulphuric acids.

Violet Hydrate and Uranic Hydrate.—On exposing an aqueous solution of uranic oxalate to the action of the solar rays, Ebelmen observed that the solution became cloudy and deposited a flocculent material of a violet-brown colour. After being washed several times with boiling water, the precipitate no longer contained oxalic acid; Ebelmen admitted that it was an intermediate hydrated oxide, U_3O_8. When thrown on a filter and exposed to the air until it was completely dried, this hydrate was converted into pure hydrated uranic oxide, UO_3,H_2O. The preparation of the violet hydrate, and consequently the hydrated uranic oxide, would thus be very simple if the low solubility of the uranic oxalate (100 parts of water dissolve 0·8 part of oxalate at 14°) did not render it impracticable.

I have tried to modify this method by using other, more soluble, salts of uranium; I was successful, more especially with the acetate. The aqueous solution of this salt is reduced by light to a certain extent; but this reaction, which is very slow, becomes, on the contrary, rapid if we cause certain organic matters to intervene, particularly alcohol or ether.

It is sufficient to expose to sunlight an aqueous solution of subacetate of uranium containing ether, to obtain a very abundant violet precipitate in a few minutes. The solution of the same salt in alcohol at 90° gives rise to an analogous phenomenon. This violet hydrate, after several washings with boiling water, no longer contains any acetic acid. It dissolves in acids giving a mixture of the uranic and uranous salts; it is therefore a hydrated uranoso-uranic oxide; it is amorphous and flocculent. I have applied the congelation method to this compound without having been able to get any definite crystalline formation, neither have I been able to obtain any satisfactory results by heating it in a sealed tube. I intend following up these experiments.

The violet hydrate, prepared by the reduction of the acetate, is less oxidisable than that of Ebelmen; it enables us, nevertheless, to obtain the pure amorphous and crystalline uranic hydrate.

Amorphous Uranic Hydrate, Crystallised Hydrate, $UO_3.H_2O$.—The uranoso-uranic hydrate is transformed with the greatest ease into uranic hydrate by oxidation in the air. It suffices to keep it moist; the uranic hydrate thus produced has the formula $UO_3.H_2O$; it is absolutely identical with that of Ebelmen. Heated to 100° with water in an open beaker, the violet hydrate oxidises and becomes crystalline. The crystals formed under these conditions belong to the orthorhombic system. When examined under the microscope they appear:—1. In rectangular orthorhombic tables, with truncations on the four angles—by suppression of the small sides some of them take a pseudo-hexagonal shape; 2. Other crystals have the appearance of rectangular orthorhombic prisms, with a principal direction of elongation. Finally, there are also observed forms in between these two types.

One of these crystals showed, in converging light, an acute bisector perpendicular to the flat face and corresponding to $n\,p$ (negative biaxis). Following the relative values of the indices, the facets which are too small to be examined by converging light showed that their flattening was perpendiular to $n\,p$, and that the negative index ($n\,m$) is nearer to $n\,g$ than to $n\,p$ (negative biaxis).

The formula of this hydrate is $UO_3.H_2O$. I found:—

	I.	II.	Calculated.
UO_2	88·6	88·5	88·9
Water	11·4	11·5	11·1

It is not decomposed in dry air at 100°.

M. Riban has already obtained a hydrate of the same formula, but crystallised in the hexagonal system, by heating a 2 per cent solution of subacetate of uranium to 175°, in a sealed tube, for 100 hours.—*Bull, Soc. Chim.*, Ser. 3, xxiii., No. 10.

EXPERIMENTS ON SOME NEW EXPLOSIVE AND DETONATING SUBSTANCES.

By U. ALVISI.

I propose passing in review a few new explosives which I have prepared, based on perchlorate of ammonium, and I will briefly describe the commercial production of the perchlorate.

The *cremonites*, so-called in honour of Prof. Cremona, are mixtures of perchlorate of ammonium, picric acid or picrates, and trinitrocresol or its salts. In this article I will only deal with the mixtures of picric acid and perchlorate of ammonium. The results I have obtained with these mixtures are as follows:—

1. The cavity produced in lead cylinders had the shape of an amphora.

2. The mixture of picric acid and perchlorate of ammonium, kept for more than a year, had undergone no alteration.

3. The mixtures which gave the best results had the following compositions:—

A.	Picric acid	2·30 parts.
	Perchlorate of ammonium	2·35 „
B.	Picrate of ammonium ..	2·46 „
	Perchlorate of ammonium	2·35 „

4. Numerous experiments have proved that the cremonites are much more powerful than picric acid alone.

5. The difference between the explosive force of picric acid and that of the cremonites is the more considerable as the quality of the picric acid used is inferior.

I expected, *d priori*, that mixtures of perchlorate of ammonium and coal would give rise to very powerful explosives possessing peculiar properties; and, as a matter of fact, this is the case, as I have proved in the course of my experiments. I have named these mixtures *cannel powder*, to distinguish them from ordinary powders, and to intimate that they are based on perchlorate of ammonium.

As for the kind of coal used, the boghead variety from Australia has given good results; but the best results were obtained with cannel coal from Scotia. The best mixture was composed of—

Cannel from Scotia	12 parts.
Perchlorate of ammonium	60 „

The method of making this new powder is much more simple than that of ordinary powder. The finely divided carbon is intimately mixed with the powdered perchlorate of ammonium in an apparatus to which a gyratory movement can be imparted. The mass obtained is slightly moistened with water containing a small quantity of gum; it is then compressed and granulated like ordinary

blasting powder, or made into cylinders, rods, &c., of a definite weight, and having an opening in the centre for the reception of the detonator.

When the powder is intended for use under water, it is coated with an impermeable varnish.

Samples of cannel powder kept for seven months do not show any alteration.

With regard to its uses, they are the same as those of dynamite, over which it has the advantage of being more powerful.

Commercial Manufacture of Perchlorate of Ammonium.—The preparation of perchlorate of ammonium is based essentially on the two following facts:—

1. The transformation of chlorate of sodium into perchlorate by heat; 2. The action of nitrate of ammonium on perchlorate of sodium in concentrated solution. We obtain a precipitate formed by small crystals of perchlorate of ammonium which only require to be submitted to the drying action of the filter-pump.

Some time previous to me, in the year 1871, Th. Schlœsing (*Comptes Rendus*, vol. lxxiii., p. 1271) had the idea of replacing the perchlorate of potassium with the corresponding sodic salt for the purpose of preparing perchlorate of ammonium. But this author had in no way the intention of utilising the substance obtained for the purpose of making explosive materials; his only idea, in its preparation, was to eventually derive perchloric acid from it, an analytical reagent used for the separation of sodium and potassium. But while I had recourse to nitrate of ammonium, Schlœsing recommended the chloride.—*Gazzetta Chimica Italiana*, 1899, ii., p. 478.

A PRELIMINARY STUDY OF THE COBALTICYANIDES.*

By E. H. MILLER and J. A. MATHEWS.

SEVERAL years ago, during the course of an investigation of the ferrocyanides of zinc and manganese (*Journ. Am. Chem. Soc.*, 1897, xix., 547), it was suggested that a study of the reactions between potassium cobalticyanide and solutions of metallic salts might be of interest, and that possibly this compound might be found useful as a reagent in either qualitative or quantitative analysis. A careful search through the literature showed that the subject had not been thoroughly investigated; that the cobalticyanides of many metals were unknown, and that the properties, especially the solubilities, of the known cobalticyanides, were but incompletely recorded. Apparently no work has been done on these compounds for many years, while most of the investigations bear dates prior to 1857.

The general procedure adopted in this work was:—

First.—To try the action of a half-normal solution of potassium cobalticyanide (based on the hydrogen equivalent in grms. per litre) on half-normal solutions of all the common metals. They were first added to each other in equal quantities and the filtrate tested to see which reagent was in excess; and, having found the amount of potassium cobalticyanide needed to precipitate a given metal, to work in subsequent experiments with quantities which gave a slight excess of the precipitant. Although these solutions were only approximately half-normal, they were made with sufficient care to distinguish in the resulting precipitates between a normal cobalticyanide and a double potassium-metallic cobalticyanide. The indications show that in nearly every case a normal salt is produced by a reaction of simple double decomposition.

Second.—The solubility of the precipitates so obtained was tested in every instance in nitric, hydrochloric, sulphuric, acetic, and oxalic acids, in ammonium hydroxide and caustic potash; in all cases both cold and hot.

These tests were made by adding the acid or alkali to the original liquid containing the precipitate, and, in each instance, an amount of solvent equal in volume to the original liquid was employed. A few precipitates were filtered, and their solubilities tested in concentrated acids.

The special solutions used were of the following strengths:—

$K_3Co(CN)_6$ (N/2)	55·52 grms. per litre.			
HNO_3 (1 : 3)	sp. gr. 1·135	= 22	per cent	
HCl (1 : 1)	,, 1·10	= 20	,,	
H_2SO_4 (1 : 4)	,, 1·20	= 28	,,	
$HC_2H_3O_2$	,, 1·03	= 22	,,	
$H_2C_2O_4$ (saturated solution)		= 10±	,,	
NH_4OH (1 : 2)	,, 0·96	= 10	,,	NH_3
KOH	,, 1·11	= 10	,,	K_2O

No highly coloured or characteristic precipitates have as yet been found which can be employed as indicators to show an excess of potassium cobalticyanide, nor do the precipitates settle readily enough to allow this point to be determined by cautious additions of cobalticyanide to the clear supernatant liquid or by spot tests. In every precipitation the potassium cobalticyanide was added to the metallic salt solution and the presence of either substance in excess had usually to be determined in a few drops of the filtrate from the resulting precipitate. The experimental part of our work will show that, in general, the cobalticyanides insoluble in water, are also insoluble in and unaffected by acids, but are soluble in or decomposed by alkalis. None of them are decomposed by boiling, as is the case with several ferricyanides. There is little similarity in either solubility or appearance between the corresponding ferri- and cobalticyanides. The metals, except those of the alkalis and alkaline earths, were taken up in their qualitative sequence, beginning with the silver-lead-mercury group.

Silver Cobalticyanide.—When equal volumes of half-normal silver nitrate and potassium cobalticyanide solutions are mixed, the silver is completely precipitated as silver cobalticyanide, a white curdy precipitate, which settles and filters well; is insoluble in all the acids used, being transposed by hydrochloric acid; is soluble in ammonia, and decomposed by potassium hydroxide giving a precipitate consisting mostly of silver oxide.

Lead Cobalticyanide.—Neither lead acetate nor nitrate are precipitated by potassium cobalticyanide, either in neutral or acid solutions. Zwenger (*Liebig's Ann. Chem.*, lxii., 158) made it from lead carbonate and cobalticyanhydric acid. This salt, crystallising in laminated crystals containing 4 molecules of water, he says, is soluble in about three parts of water, from which solution it is precipitated by ammonia as a basic salt.

Mercurous Cobalticyanide.—Mercurous nitrate gives with potassium cobalticyanide a white flocculent precipitate, which settles quickly. It was therefore possible to use concentrated ammonia as an indicator. Spot tests on porcelain showed a black precipitate as long as there was an excess of mercurous nitrate. Mercurous cobalticyanide is transposed by hydrochloric acid, and seems to be partly changed by hot sulphuric acid, but not by oxalic acid. It is unaffected by nitric and acetic acids, but decomposed by alkalis. Mercurous cobalticyanide is probably a normal salt having the formula $Hg_3CO(CN)_6$.

Mercuric Cobalticyanide is unknown. There is no precipitate formed when potassium cobalticyanide is added to mercuric chloride either in neutral or acid solution.

Arsenic, in hydrochloric, sulphuric, or ammoniacal solution gives no precipitate.

Antimony, in a solution sufficiently acid to prevent the precipitation of a basic salt, gives no precipitate.

Stannous salts in neutral, acid, or potassium hydroxide solution are not precipitated by potassium cobalticyanide. Gmelin ("Handbook of Chemistry," 1852, vol. vii., p. 495) states that stannous but not stannic salts are precipitated,

* Contributions from the Havemeyer Laboratories, Columbia University. From the *Journal of the American Chemical Society*, vol. xxii., No. 2.

SOLUBILITIES OF THE COBALTICYANIDES.

$K_3Co(CN)_6$.	(1). HNO_3.	(2). HCl.	(3). H_2SO_4.	(4). $H.C_2H_3O_2$.	(5). $H_2C_2O_4$.	(6). NH_4OH.	(7). KOH.	Remarks.
$+AgNO_3$, white curdy precipitate	(cold) *i* (hot) *i*	AgCl AgCl	*i* *i*	*i* *i*	*i* *i*	Partly *s* *s*	Ag_2O Ag_2O	Precipitation complete. Settles and filters well. (6) White crystalline precipitate (double salt) on evaporation. Easily soluble cold concentrated $NH_4(OH)$.
$+Hg_2(NO_3)_2$, white flocculent precipitate	*i* *i*	Hg_2Cl_2 Hg_2Cl_2	*i* *i*	*i* *i*	*i* *i*	$NH_2Hg_2NO_3$ $NH_2Hg_2NO_3$	Hg_2O Hg_2O	(3) Seems slightly soluble hot or else is partly changed to Hg_2SO_4. (5) Not changed to oxalate.
$+CuSO_4$, turquoise-blue precipitate	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*s* *s*	CuO CuO	Filters well. (6) Intense blue solution, giving double NH_3 compound on evaporation. (7) Turns green, getting darker until black CuO separates.
$+Bi(NO_3)_3$, dense white precipitate	*i* *i*	Easily *s* *s*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	$Bi(OH)_3$ Bi_2O_3	Precipitation done in strongly acid solution to prevent basic salts upon dilution. (6) Not transposed to $Bi(OH)_3$.
$+CdCl_2$, white amorphous precipitate	*i* *i*	*i* *s*	*i* *i*	*i* *i*	*i* *i*	*s* *s*	$Cd(OH)_2$ $Cd(OH)_2$	Filters badly. (2) precipitates again on cooling.
$+Fe_2Cl_6$, green, becoming yellow	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	$Fe_2(OH)_6$ $Fe_2(OH)_6$	$Fe_2(OH)_6$ $Fe_2(OH)_6$	(5) When freshly precipitated and greenish it is readily soluble in oxalic cold. Ammonia salts in quantity prevent precipitation and excess of $K_3Co(CN)_6$ retards it.
$+FeSO_4$, faintly yellowish white precipitate	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	Partly $Fe(OH_2)$ $Fe(OH_2)$	$Fe(OH)_2$ $Fe(OH)_2$	(1, 2, and 3) On boiling oxidise more or less to yellow ferric cobalticyanide. (6 and 7) Some oxidation to dark coloured ferroso-ferric compounds.
$+MnCl_2$, white precipitate	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	$Mn(OH_2)$ and $Mn_2O_2(OH)_2$	$Mn(OH)_2$ and $Mn_2O_2(OH)_2$	(1, 2, 3, 4, and 5) Seem very slightly soluble hot.
$ZnSO_4$, dense white precipitate	*i* *i*	*i* *i*	*i* Trace *s*	*i* *i*	*i* *i*	Easily *s* *s*	Easily *s* *s*	(4 and 5) Bump violently on boiling.
$+COCl_2$, rose pink precipitate	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	Partly *s* Partly *s*	$Co(OH)_2$ $Co(OH)_2$	(6) Solubility increased by ammonium salts. Almost completely soluble in hot concentrated NH_4OH. (7) At first blue basic salts, turning again pink.
$+NiCl_2$, robin's egg blue precipitate	*i* *i*	*i* *i*	*i* *i*	*i* *i*	*i* *i*	Blue sol. *s* *s*	$Ni(OH)_2$ $Ni(OH)_2$	Very voluminous precipitate, which filters poorly.
NH_4, Li, Na, Mg, Ba, Ca, Sr, Pb, Hg'', $Sn''+^{IV}$, Cr, Al, Pt, Au, Zr, Ti, Mo salts	No precipitates.							Solutions of salts of other metals are not precipitated by $K_3Co(CN)_6$ and other known cobalticyanides are easily soluble in water. Such cobalticyanides are usually made from an oxide or hydroxide acted upon by $H_3Co(CN)_6$.

but does not give the conditions under which he obtained the precipitation.

Cupric Cobalticyanide.—When a solution of copper sulphate is completely precipitated by potassium cobalticyanide solution, there results a turquoise-blue compound. This salt, apparently $Cu_3Co_2(CN)_{12}$, is insoluble in all acids, cold or hot; very soluble in ammonia, and with caustic potash it turns green, becoming darker and darker until black cupric oxide separates. The ammoniacal solution upon evaporation gives small shining blue crystals, to which Zwenger (*loc. cit.*), who first worked with this compound, assigns the formula $Cu_3Co_2(CN)_{12}.2NH_3.5H_2O$.

Cuprous Cobalticyanide, of which no mention is found in the literature, results from the precipitation of cuprous chloride by means of potassium cobalticyanide. It is a faintly yellow precipitate, which filters badly; it is insoluble in acids and cold ammonia, but soluble in hot dilute ammonia. It is partly decomposed by potassium hydroxide in the cold, and completely when hot. It is slowly oxidised by nitric acid, giving the blue cupric cobalticyanide.

Bismuth Cobalticyanide.—A solution of bismuth nitrate, rather strongly acid with nitric acid, is precipitated by potassium cobalticyanide, giving a dense white precipitate, very soluble in hydrochloric but insoluble in the other acids and ammonia. Potassium hydroxide gives in the cold $Bi(OH)_3$, which, on boiling, may be changed to yellow Bi_2O_3. The original precipitate seems to be a normal cobalticyanide, but it has not yet been analysed, and no mention of such a compound was found in the literature.

Cadmium Cobalticyanide.—The statement appears in Gmelin's "Chemistry" (1852), that with cadmium sulphate, potassium cobalticyanide forms a brown precipitate, turning white later, soluble in excess of cobalticyanide and also in acids. In our experiments cadmium chloride was employed; the precipitate was white and amorphous, not soluble in excess of cobalticyanide, nor in any acid except warm hydrochloric acid, from which it re-precipitates on cooling. To determine whether it is changed or not during this treatment will require quantitative work. It is completely soluble in cold ammonia, but potassium hydroxide decomposes it, giving cadmium hydroxide.

Solutions of gold, platinum, titanium, vanadium, uranium, or zirconium are not precipitated by potassium cobalticyanide.

Aluminum chloride and chromium salts gave no precipitate with cobalticyanide. Certain double ammoniochromic cobalticyanides have been described by Christensen (*J. Prakt. Chem.* [2], xxiii., p. 52) and by Braun (*Liebig's Ann. Chem.*, cxxv., 153, *et seq.*).

Ferric Cobalticyanide.—When potassium cobalticyanide is added to a solution of ferric chloride, no precipitate forms at first, but the solution assumes a light green tint, and on standing a few minutes becomes cloudy and iridescent. The precipitation increases with the formation of an amorphous yellow precipitate which runs through the filter. By boiling, it becomes canary-yellow and flocculent, filtering fairly well. The precipitation does not seem to be complete; it is retarded by the presence of an excess of cobalticyanide or by large quantities of ammonium salts. The precipitate once formed is not soluble in cobalticyanide solution, nor in mineral acids, cold or hot, dilute or concetrated. Hydrochloric acid changes the colour of the precipitate, and renders it almost impossible to filter. Insoluble in acetic acid, cold or hot; when freshly precipitated it is instantly soluble in oxalic acid, but re-precipitates on boiling. It seems that at least two different compounds are produced; they differ both in colour and solubility; the greenish one is soluble in oxalic acid, and the yellow one insoluble. The latter is formed by long standing in the cold, or rapidly by heating. Both give ferric hydroxide with ammonia or potassium hydroxide in the cold. If the ferric salt and the cobalticyanide solutions are mixed hot, precipitation of the yellow compound is instantaneous, no matter which reagent is in excess. The well-washed ferric cobalticyanide gives no red colour with ammonium thiocyanate.

Ferrous Cobalticyanide.—Ferrous sulphate and potassium cobalticyanide seem to give a normal ferrous cobalticyanide,—a slightly yellow, amorphous, slow-settling precipitate. When an excess of cobalticyanide is present there is no oxidation or change of colour in the ferrous cobalticyanide, even upon long boiling. The precipitation is complete and filters well. With nitric acid, cold and hot, and with hydrochloric and sulphuric acids, hot, there is oxidation. The precipitate darkens in varying degrees, most with nitric and least with sulphuric acid. After treatment with these acids the precipitate filters badly. No change is noticed with acetic or oxalic acids; in the presence of the latter it filters very well. With ammonia the precipitate is partially decomposed in the cold, becoming bluish, and by boiling is completely converted into ferrous hydroxide. With potassium hydroxide it gives a greenish tint, darkening on standing, becoming slate-coloured by boiling, forming probably a mixture of ferrous and ferroso-ferric hydroxides.

Manganese Cobalticyanide.—This precipitate, apparently normal, is pure white and filters well. It is insoluble in all cold acids, but not absolutely so in hot mineral acids. Alkalis decompose it, giving first $Mn(OH)_2$, and later, by oxidation, $Mn_2O_2(OH)_2$.

Zinc Cobalticyanide is formed when a solution of zinc sulphate is precipitated by potassium cobalticyanide. It is pure white, settles quickly, and filters well; insoluble in hot water, cold or hot acids, except that a trace seems to dissolve in hot sulphuric acid. This precipitate as well as a few others, *e.g.*, ferrous and ferric cobalticyanides, on boiling with hydrochloric acid passes into a condition which renders it impossible to filter even on double filters. It is very soluble in alkalis.

Cobalt Cobalticyanide.—From solutions of cobaltous chloride, potassium cobalticyanide precipitates a rose-pink, amorphous, rather gelatinous precipitate which is insoluble in cold and hot acids. At 100°, or even lower, it loses water and becomes blue. This fact was noted by Zwenger, who also states that he formed this compound by heating cobalticyanhydric acid with concentrated sulphuric acid and diluting with water before the decomposition was complete. A peculiarity of many cobalticyanides is the large amount of water which they retain after drying at 100° C. Zwenger noticed that the blue anhydrous cobaltous-cobalticyanide rapidly absorbs moisture from the air, and that when water is poured upon it there is a considerable evolution of heat. We found that the ferrocyanides of zinc and manganese acted in the same way. When cobaltous cobalticyanide is treated cold with ammonia, it gives a brown solution and pink residue; on boiling, the solution becomes pink and the residue greenish-brown. The presence of ammonium salts increases the solubility of this compound in ammonia; it is completely soluble in concentrated ammonia. Caustic potash gives first blue basic salts, and on standing or boiling rose-coloured cobaltous hydroxide.

Nickel Cobalticyanide results when potassium cobalticyanide is added to solutions of nickel salts. Zwenger states that this precipitate cannot be washed free from potassium salts, and that the pure nickel cobalticyanide, $Ni_3Co_2(CN)_{12}$, must be made from cobalticyanhydric acid and a nickel salt. The precipitate is robin's egg blue in colour, very voluminous, and dries up to a vitreous mass with conchoidal fracture. It is insoluble in water and acids, cold or hot; soluble completely in ammonia, cold or hot. Potassium hydroxide gives instantly pale green nickel hydroxide.

The effect of the reagents used on the precipitates formed by potassium cobalticyanide is shown by the accompanying table. A glance at this tabulation will show several possibilities for new separations:—

1. The lead salt is exceedingly soluble, while those of silver, copper, and bismuth are insoluble in either water

or nitric acid. This may find an application in the analysis of pig lead, when the separation of relatively small quantities of these impurities would be much more preferable to the separation of the lead as sulphate. The separation of the bismuth can be effected either by treatment with hydrochloric acid, which dissolves the bismuth cobalticyanide and leaves the copper as cobalticyanide and the silver as chloride, or by ammonia which leaves the bismuth and dissolves the copper and silver. The bismuth compound, if not suitable for weighing, can be decomposed by potassium hydroxide and weighed as Bi_2O_3, or one of the new volumetric methods may be employed for its estimation.

2. As the precipitation of ferric cobalticyanide in the cold can be completely prevented by the presence of ammonium sulphate, and probably by other compounds, it is possible to precipitate zinc, manganese, nickel, or cobalt in the presence of iron. With zinc ores containing iron and manganese, the manganese and zinc can be precipitated as cobalticyanides, and then separated by treatment with potassium hydroxide, which readily dissolves the zinc cobalticyanide and leaves the manganese as $Mn_2O_2(OH)_2$.

Another possible application is in the analysis of nickel steel, where the separation from ferric iron, if sufficiently complete, would be more convenient than the method in use, based on the solubility of ferric chloride in ether. The nickel cobalticyanide after filtration can be converted into hydroxide and then dissolved in acid for the cyanide titration or for electrolysis.

These and other possible uses of the cobalticyanides are under investigation at Columbia University.

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, June 8th, 1900.

Dr. J. H. GLADSTONE, F.R.S., Vice-President, in the Chair.

A PAPER ON "*The Magnetic Properties of Alloys of Iron and Aluminium*" (Part II.), by S. W. RICHARDSON and L. LOWNDS, was read by Dr. RICHARDSON.

Experiments have been made to ascertain in what way the hysteresis loss between given limits of the field strength is connected with the temperature for an alloy containing 3·64 per cent of aluminium. The experiments show that the hysteresis loss attains a maximum value at a temperature considerably higher than the temperature of maximum induction. The changes produced in the magnetic properties of the alloy by heating and subsequent cooling have also been investigated. The properties depend largely on the previous history of the specimen, but there does not appear to be any essential difference between the behaviour of the alloy during heating and cooling (except near the temperature of minimum permeability). Experiments have also been conducted on the abrupt change in the permeability that takes place at a temperature of about 650° C. The conclusions arrived at are as follows:—(1). The hysteresis loss at first diminishes as the temperature rises. It then increases, and reaches a maximum at about 550° C. On further heating it falls off rapidly, and is negligible at 700° C. (2). The magnetic properties of the specimen depend largely on its previous history. (3). There is no essential difference between the behaviour during heating and cooling except near the temperature of minimum permeability. (4). An abrupt increase in the permeability takes place at about 650° C. during heating, followed by an equally abrupt diminution on further heating. (5). This abrupt change is more marked with falling than with rising temperatures. (6). Continued heating and cooling diminish the permeability. (7). The curve connecting temperature of minimum permeability and percentage of aluminium is a straight line. (8). The microscopic examination of the specimens shows the presence of crystals.

Prof. S. P. THOMPSON asked if the specimens had been kept for any length of time at a high temperature, because crystals changed and grew in metals at temperatures even far below their melting-points.

Prof. REINOLD asked if any specimens had been examined where the crystalline structure had not been observed.

Mr. BLAKESLEY asked if any explanation of the orientation of the crystals could be given.

The CHAIRMAN said it was difficult to know exactly what substances were being dealt with. They might be pure alloys, or mixtures of two or three alloys with iron or aluminium.

Dr. RICHARDSON, in reply, said the crystals might be dissolved in nitric acid and analysed, but at present he did not know their composition.

Mr. W. CAMPBELL then read a "*Note on Crystallisation produced in Solid Metal by Pressure.*"

In the preparation of sections of tin, particles cling to the file, and, if allowed to remain, tend to tear the surface of the metal. The effect is not immediately noticeable, but on etching the polished surface there appear, besides the usual structure of the tin, lines of much smaller crystals with irregular boundaries, but possessing different orientation. The effect is only superficial because it can be removed by polishing. The same behaviour is noticed in some alloys, and it would thus appear that the pressure of a file is sufficient to cause a metal or an alloy to rearrange itself.

Prof. S. P. THOMPSON suggested that the effect might be due to local heating caused by tearing rather than to pressure.

Mr. CAMPBELL said the effect was not due to the heating of the file, because if the file were kept perfectly clean no crystals formed.

Prof. S. P. THOMPSON asked if scratching the surface with a diamond produced crystallisation.

The author said he had tried with a sharp knife without success, but cutting with a blunt chisel produced crystallisation along the chisel mark.

A paper on "*The Viscosities of Mixtures of Liquids and Solutions*" was read by Dr. C. H. LEES.

Three formulæ have been suggested for expressing the viscosity of a mixture in terms of the percentages and viscosities of its constituent parts. The first of these represents the viscosity as being the sum of a number of terms, each one of which is the product of the percentage of any constituent and its viscosity. The second formula represents the logarithm of the viscosity in a similar manner, and the third one the reciprocal. None of these formulæ represent the viscosity of a mixture with closeness. The author suggests a formula in which the mth power of the reciprocal of the viscosity of a mixture is equal to the sum of a number of terms, each one of which is the product of the percentage of any constituent, and the mth power of the reciprocal of the viscosity of that constituent. This formula gives satisfactory agreement, and moreover leads to Slotte's formula for the variation of viscosity with temperature.

The SECRETARY read a note from Prof. WOOD on "*An Application of the Method of Striæ to the Illumination of Objects under the Microscope.*"

The object chosen was powdered glass immersed in cedar oil of the same refractive index. The glass particles were almost invisible under ordinary conditions of illumination. The illuminating system was then arranged as follows:—A screen bounded by a straight edge was placed in front of an incandescent gas lamp so as to cut off half of the mantle, and give a source of light bounded by one perfectly straight edge. A small lens of very short focus was placed below the stage as close as possible to the

object. The lamp was at a distance of six feet, and the light reflected from the mirror was brought to a focus by this lens, passing through the object on its way. An image of the lamp was formed in space and viewed by the microscope. A little strip of thin brass with a carefully cut straight edge was fastened to the stand carrying the bull's-eye condenser, and moved into position between the objective and object, so as to cut off the flame image with the exception of a narrow thread of light along the straight edge. The brass screen must be in the plane of the flame image with its edge parallel to the straight edge of the flame. The brass was then advanced over the flame until nearly all the light was cut off. Upon lowering the microscope until the object was in focus, and carefully advancing the brass strip until practically all the flame image was cut off, it was found that the glass particles suddenly appeared with great sharpness, showing as distinctly as if in air. Two photographs of glass in oil were shown, one taken with ordinary illumination, and the other by the Schlieren-Methode.

The meeting then adjourned until June 22nd.

ROYAL INSTITUTION.

General Monthly Meeting, June 11th, 1900.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE following were elected Members:—Mr. C. E. Baxter, Mr. C. Coward, Mr. A. Dupré, Mr. L. V. Harcourt, Mr. W. C. Prescott, and Mrs. M. F. Thorne.

The special thanks of the Members were returned to Mr. Harold Swithinbank for his donation of £50 to the fund for the Promotion of Experimental Research at Low Temperatures.

The Managers reported, that at their meeting held this day the following Resolution was unanimously agreed to:—

"The Managers of the Royal Institution of Great Britain, on the occasion of the retirement of Sir Frederick Bramwell from the office of Honorary Secretary, desire to place on permanent record an expression of their high appreciation of the admirable way in which he has performed the duties of that office and of his signal services to the Institution generally.

"Elected a Member of the Royal Institution in 1876, Sir Frederick Bramwell has since then delivered seven Friday Evening Discourses on subjects cognate to that branch of applied science with the progress of which in this country, during the Victorian Era, his name must ever remain honourably associated.

"Having joined the Board of Managers in 1879, he was induced, in 1885, notwithstanding professional engagements of the most onerous and responsible character, to undertake the additional burden of the duties of Honorary Secretary to the Institution. For fifteen years these duties have absorbed no inconsiderable proportion of his time, and have been discharged with incomparable energy, business ability, and courtesy. Himself a generous patron of the Institution, and foremost to support every project for its advantage, he has been able to suggest improvements in the administration of its property which have added to its material resources. Mainly concerned in the arrangement of the courses of Lectures and Friday Evening Discourses, he has succeeded with no small expenditure of labour in maintaining these at a high level of educational value, and in making them attractive and popular and representative of every modern advancement in the arts and sciences. While extending the usefulness of the Institution in every direction, and introducing into it many new members, he has by his genial personality done much to promote smoothness and harmony of working in its several departments.

"The Managers feel that the Royal Institution has been singularly fortunate in having so long enjoyed the services of Sir Frederick Bramwell in the capacity of Honorary Secretary, and they rejoice to know that although he is no longer to fill that office, they are still to have the benefit of his counsels at their Board. Sir Frederick Bramwell's name is indelibly stamped upon the history of the Royal Institution for the last quarter of the nineteenth century. He will always be gratefully remembered by its Members, but the Managers wish to add to personal remembrance this formal record of their cordial recognition of his merits."

The CHAIRMAN announced that a desire had been expressed that a bust of Sir F. Bramwell should be placed in the Royal Institution.

NOTICES OF BOOKS.

Richter's Organic Chemistry, or Chemistry of the Carbon Compounds. Edited by Prof. R. ANSCHÜTZ. Authorised translation by EDGAR F. SMITH, Professor of Chemistry University of Pennsylvania; third American from the eighth German Edition. Vol. II. *Carbocyclic and Heterocyclic Series.* With illustrations. London: Kegan Paul, Trench, Trübner, and Co., Limited. 1900. Pp. 671.

THE first volume of this work was noticed in this column on March 2, 1900, and in a general sense we have nothing to add to the remarks we then made in regard to the general excellence of the book.

Volume II. deals with the compounds of the carbocyclic and the heterocyclic series. The first of the above series comprises the tri-, tetra-, penta-, and heptacarbocyclic compounds, which are disposed of in a few pages; these are followed by the large and important group of hexacarbocyclic compounds, to which considerably more than half the volume is devoted. We next come to Part III. on the heterocyclic compounds, comprising the three, four, five, and six numbered heterocyclic substances.

In our notice of Vol. I. we referred to the excellent index of 36 pages; Vol. II. is even better equipped in this respect, the index consisting of no less than 65 pages of two columes each, which, we read, is due to the patient work of Messrs. W. Taggart, O. L Shinn, and J. B. Moyer.

Field Testing for Gold and Silver. By WM. HAMILTON MERRITT, F.G.S., &c. London: Crosby Lockwood and Son. 1900. Pp. 144.

As a practical man the author soon found that the elaborate blowpipe sets prepared for colleges were much too complicated for the field, and in this practical manual for miners and prospectors he has shown how good results can be obtained with very cheap and simple appliances.

Field testing takes up about half the pages of this book, and comprises all the necessary instructions for sampling, panning, the use of the blowpipe, ivory-scale, and more complete assays with the field furnace. The second part deals with practical mineralogy, geology, and the diagnostic characters of rocks. Part III. contains a glossary of useful mining terms. We must, however, warn our readers against a too free use of such terms; they are often used to disguise ignorance, and will frequently deceive the unwary investor, who is apt to think that a man using such an excess of technical language must necessarily have had great experience.

This pocket-book is of a convenient size, well bound, and illustrated with nine plates from photographs, and numerous figures in the text.

A Pocket-book for Chemists, Chemical Manufacturers, Metallurgists, Dyers, Distillers, Brewers, Sugar Refiners, &c. By THOMAS BAYLEY, Assoc. R.C.Sc.I. Seventh Edition. Revised and enlarged. London: E. and F. N. Spon, Limited; New York: Spon and Chamberlain. 1900. Pp. 559.

BAYLEY'S Chemists' Pocket-book is so well known that it needs but few words to remind our readers of its excellent character. The present edition has been re-arranged, and to a great extent re-written, and shows a marked contrast to the first edition, published in 1878, which consisted of 421 pages only. This pocket-book is without doubt the best of its kind, and we hope the author will be as successful in the future as he has been in the past.

Arithmetical Exercises in Chemistry. By LEONARD DOBBIN, Ph.D. With a Preface by Professor CRUM BROWN. Third Edition. Edinburgh: James Thin; London: Simpkin, Marshall, and Co., Limited. 1899. Pp. 52.

THE usefulness of this book may be gauged by the fact that a third edition is now required. The general plan of the work remains unaltered, but the opportunity has been taken to introduce, in the form of appendices, the Denominations of the New Metric Standards, and the Tables of Imperial and Metric Equivalents adopted by the Board of Trade, and approved by orders in Council, May 19, 1899. A few other changes have been made, and additional exercises included.

The Arithmetic of Chemistry. By JOHN WADDELL, B.Sc. (Lond.), Ph.D. (Heid.), D.Sc., (Edin.). London and New York: Macmillan and Co., Limited. 1899. Pp. 133.

IN these pages the author gives a simple treatment of chemical calculations with a view to smoothing away the difficulties encountered by students in this direction. In preparing for examinations, as well as in studying chemistry with a view to retaining knowledge, students will obtain great assistance from this book, as the points dealt with are very clearly expressed; in this connection we may mention the manner in which the author puts forward the grm.-molecular volume of gases, a subject which has been a stumbling block to many beginners.

The Splitting-up and the Properties of Phenylglycolic Acid.—E. Rimbach.—Phenylglycolic acid has been split up by M. Dewkowitsch; according to the author, the following are the best conditions for effecting this decomposition:—60 parts of the acid, and 120 parts of crystallised cinchonine are dissolved in 3 litres of water, and heated for an hour on the water-bath; then, after standing for twelve hours, it is filtered and crystallisation started by dissolving a certain quantity of chloride of sodium in the liquid. In this manner about 100 parts of the right-handed salt are obtained; this is purified by crystallisation in boiling water. Right-handed phenylglycolic acid crystallises in hemimorphous clinorhombic prisms. Its rotatory power in aqueous solutions increases considerably with dilution ($143{\cdot}7°$ for 0·058 per cent). Its rotatory dispersion is normal and fairly strong. Inactive synthetic phenylglycolic acid constitutes a racemic, and not a mixture. The left-handed phenylglycolate of cinchonine which remains in the mother-liquor crystallises in hemihedric rhombic tables. Its rotatory power in aqueous solution (at 2·33 per cent), is $[\alpha]_D = +92{\cdot}1°$; that of the right-handed salt is $[\alpha]_D = +152{\cdot}4°$, for a concentration of 2·43° per cent. The author has determined the solubilities in water of the two salts, and of the right-handed acid at different temperatures.—*Berichte*, xxxii., p. 2385.

CORRESPONDENCE.

THE ADVANTAGES OF UNIFORMITY IN MANURE ANALYSIS.

To the Editor of the Chemical News.

SIR,—Referring to the letter from one of us *re* "Uniformity of Chemical Manure Analysis" (CHEMICAL NEWS, vol. lxxxi., p. 143), it might be interesting and instructive to point out the advantages that would result from that uniformity for all interested parties.

As was already shown, there would be an enormous advantage for the manufacturer. As things are at present, there is constant cause for disagreement by discrepancies, rude surprises by too low tests, &c.; and the chemical analysis, instead of being a trustworthy guide for him, will very often only confuse him. Say, for instance, a public chemist gives a totally different analysis of a sample than his own works official had certified. To make sure of the test, he sends the sample to a third chemist, with the result that perhaps he has to choose now between three different results; or, say, he believes the test of the second chemist, which may show too low, and alters his mixing according to his test; he may find that the next sample from this altered mixing, tested by another referee chemist, would show a result far too high, thereby showing great waste in materials if he has to take this latter test as being correct.

Such things could never occur if uniformity of methods would be introduced; the chemical analysis would then be a really true guide for the manufacturer. One chemist would find the sample to be much the same as another, if their separate analyses were correctly performed. If a manufacturer wants to deliver a really good article, he knows exactly how to regulate his mixings accordingly, and need—as at present he has—have no fear of finding his manures condemned as being below guarantee, simply by the discrepancies due to want of uniformity.

On the other hand, those less scrupulous manufacturers who purposely try to take advantage of the confusion caused by the very varying analyses furnished by different chemists, would soon have their sharp practice brought to light and effectually stopped.

And this is where the farmer would greatly benefit. It is to his interest, chiefly, that the manure trade should be firmly fixed on fair and honest lines; and that he can have an absolutely sure guide, through the analysis, for finding the fair dealing firms, and thus protect himself against would-be fraudulent dealers.

With more confidence he will then see in the public chemist his indispensable never-failing adviser, and more than ever will he seek his advice; there will be more work for the public chemists instead of less, as some of them may fear, and the public generally will have a much higher opinion of the profession than possibly it may have at present. Now it may often be heard in industrial circles of a "buyer's" chemist and a "seller's," chemist, in that sense that the first is thought to try to favour the buyers by his relatively low, the latter the sellers by his relatively high, results. For the sake of the honour of the analytical chemists of Great Britain and Ireland, we are much inclined to attribute these divergent results, in nearly every case, to the want of uniformity, and only in exceptional ones to direct and intentional fraud. Knowing as we do the different results arrived at by different methods, and knowing also that most probably a chemist keeps to a method once it has found favour in his sight, may explain partly why certain members of the profession are frequented by "buyers" and others chiefly by "sellers," because these two soon find out the one who, in their eyes, gives the best results.

If, then, uniformity was introduced, such terms as mentioned above would be speedily dropped, assuming of course that it is the want of uniformity from which such

things spring; and, on the other hand, if there were any impure intentions on the chemists' part, they also would be most effectually checked, as a too high or too low result could then be very easily proved to be incorrect.

As regards the works' chemist, each will know himself well enough how much bother, annoyance, useless work, and waste of time uniformity would save him. The manufacturers and managers are only too much inclined to make their works' chemists responsible for discrepancies, and indeed it is only natural, if these appear over and over again; and yet even the greatest care and over-anxious accuracy on the part of the works' chemist cannot help it, if the real source is not removed.—that is, want of uniformity.

Therefore *Uniformity* in the *Methods of Chemical Manure Analysis* would be highly beneficial to all parties concerned. Still, in spite of this, nothing has been done in the matter of this great reform, though there is, to our mind, more than one way possible.

The greatest and most widely-reaching help, of course, would come if the government took the matter into their own hands. If that were impossible, the public chemists have sufficient power to bring about this desirable reform by agreeing amongst themselves upon some certain fixed method for analysis of manures, and making that method rigidly official. If that were done, we have not the slightest doubt but that it would soon be adopted by the manufacturers; and the opportunity also arises for the manufacturers to set the ball of reform in this matter rolling, if the bigger firms adopted any particular method and only bought and sold on the results furnished by that method.

In Germany the matter has been settled by the "Control und Versuchsstationen," and in other European states, also in the United States, arrangements of one kind or another have been made. Why, then, should the United Kingdom be left behind in the endeavour to make this most important branch of chemical manufacture as near perfection as possible.

A. YOUNG.
W. JERWITZ, Ph.D.

Dublin, June 6, 1900.

SYNTHESIS OF ARSENIC.

To the Editor of the Chemical News.

SIR,—The remarks of "Analyst" in criticism of my note that "there is evidently an error in the figures" given by Prof. Fittica, are not justifiable. If 0·501 grm. phosphorus yields 0·2012 grm. arsenic sulphide, the percentage of arsenic would be 24·4, and not 2·5 as stated in the German original. The figures, therefore, are *not* "perfectly correct."—I am, &c.,

THE TRANSLATOR.

June 9, 1900.

SYNTHESIS OF ARSENIC.

To the Editor of the Chemical News.

SIR,—In reply to "Analyst's" letter of June 8th, I would beg to point out, what I should have thought would have been evident to most readers, that "there is evidently an error in these figures." If 0·501 phosphorus is equivalent to 0·2012 As_2S_3, then the percentage of As is 24·49, and not 2·5 as given in the paper, thus confirming the translator's remark.—I am, &c.,

ANOTHER ANALYST.

London, S.W., June 9, 1900.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—May I inform the gentleman who has written to you *re* the above, and whose letter was published in CHEMICAL NEWS, vol. lxxxi., p. 263, that the device he mentions was well known to alchemists when I was on the earth.—I am, &c.,

A GHOST OF 200 YEARS.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. CXXX., No. 20, May 14, 1900.

Hydrated Peroxides of Calcium.—M. de Forcrand.—Mixtures of solutions of hydrogen peroxide and the base show that lime forms, like baryta and strontia, precipitates of hydrated peroxide more soluble than those of the peroxides of strontium, but less soluble than the corresponding barium salts. Between 10° and 16° baryta and strontia give precipitates containing 8 or 9 molecules of water, whilst the calcium precipitate contains only 2 molecules of water, and has a very different heat of formation. The transformation of calcium dioxide into the hydrated state depends solely on the temperature; the critical temperature being somewhere about 15—20°.

Allotropic Transformation of Alloys of Iron and Nickel.—L. Dumas.—Dewar and Fleming showed that when an alloy of nickel and iron containing 29·07 per cent of nickel is immersed in liquid air, a non-reversible transformation takes place. This particular alloy begins to undergo the reversible transformation above 0°. Carbon has the property of lowering the point of non-reversible transformation. The author continues the study of this alloy, and finds that nickel lowers the points of transformation of iron, and iron those of nickel.

Preparation of some Aluminium Compounds and their Corresponding Hydrogen Derivatives.—M. Fonzes-Diacon.—The author describes a rapid and novel method of preparing aluminium sulphide by the action of sulphur vapour on aluminium filings. He also obtains aluminium selenide in the pure state, and was able to prepare a phosphide and arsenide of aluminium—bodies which had not previously been obtained. Antimony will also combine with aluminium, but with great difficulty. Finally, the author describes the means of obtaining, owing to the easy decomposition of the above compounds by water, large quantities of SH_2, SeH_2, PH_3 and NH_3; also in the same way, but in less quantity, antimony hydride.

Estimation of Thallium.—V. Thomas.—The method for the estimation of thallium suggested by the author is based on the transformation of the salts of the protoxide into the salts of the peroxide, but it has the advantage of extreme simplicity and of a greater accuracy than the old methods. Bromide of gold is the oxidising agent used—or, more exactly, bromo-auric acid. By reduction, the gold bromide loses 3 atoms of bromine and gives a metallic deposit of gold, which can be weighed. When the reduction is ended, it is found that 2 atoms of gold correspond to 3 atoms of thallium.

Action of Anhydrous Aluminium Chloride on Acetylene.—E. Baud.—Pure dry acetylene is completely absorbed by aluminium chloride, even in the cold, when in a closed vessel. If heated to 70°, absorption is total in some minutes. A different reaction occurs when a current of acetylene is passed over the hot chloride. The chloride at first becomes heated, then, when the temperature reaches 70°, vapours are produced, and a reddish sublimate forms, which finally becomes black. The gaseous current is almost all absorbed, and there is a considerable

development of heat. The temperature is then raised to 120—130°, and the current of gas continued until there is no increase in weight of the sublimate. The two products formed have been analysed, and apparently have the composition $(C_{20}H_{15})_7.Al_2Cl_6$ and $(C_{10}H_{15\cdot6})_7.Al_2Cl_6$.

Some New Organo-metallic Compounds of Magnesium, and their Application to the Synthesis of Alcohols and Hydrocarbides.—V. Grignard.—Methyl iodide attacks magnesium very slowly in the cold, but when a little anhydrous ether is added to the iodide a lively reaction takes place, a clear slightly coloured liquid results, with practically no deposit; this is a solution of the magnesium compound in ether, and contains one atom of magnesium dissolved in one molecule of methyl iodide. The author prepares also a certain number of secondary and tertiary alcohols. With the fatty halogen ethers the results are excellent.

Santalenes and Santolols.—M. Guerbet.—In a previous paper the author showed that the essence of oriental Indian santal contains, amongst other products, two isomeric carbides of formula $C_{15}H_{24}$, the α and β santalenes, and that the alcoholic portion probably consists of the two corresponding alcohols, the α and β santalols $C_{15}H_{26}O$. The present paper contains an account of experiments which led to the confirmation of the latter fact and the properties of the different products in a more perfect state of purity.

Tyrosinase.—C. Gessard.—Tyrosinase oxidises tyrosine with production of the red colour, which is also the effect of oxidation by a chemical agent (Millon's reaction). The blackening, as also the formation of the black precipitate, is a secondary phenomenon, depending on the elements which accompany diastase under natural conditions, and which pass with it into the solutions; this black colour can now be obtained at will.

MISCELLANEOUS.

Nitration of Benzoic Acid and of its Ethers.—A. E. Holleman.—The author has improved on the method previously given for the estimation of the component parts of a mixture of the ortho-, meta-, and para-nitrobenzoic acids, and has applied it to the study of the nitration of benzoic acid. The results are given briefly in the following table:—

	Nitration at—		
	−38°.	+8°.	+38°.
Ortho acid	14·4	18·5	22·3
Meta „	85·0	80·2	76·5
Para „	0·6	1·3	1·2

Strange bodies are not formed. The author has also studied the nitration of the benzoates of methyl and ethyl; he has observed that in this case also we obtain a mixture of the three isomers.—*Rev. Trav. Chim. P.-B.*, xviii., p. 267.

Preparation of Formose by means of Hydrated Oxide of Lead.—C. A. L. de Bruyn and A. van Ekenstein.—Heat for an hour on the water-bath a mixture of 500 c.c. of formol (at 40 per cent), 5 litres of water, and 20 grms. of hydrated oxide of lead; evaporate, and take up with a mixture of ethylic and methylic alcohols, and precipitate the salts of lead formed by the addition of ether; filter, and distil. The hydrated oxide of lead is prepared by precipitating the basic acetate with potash, and drying at the ordinary temperature after thorough washing. If we precipitate with NH_3, the oxide obtained possesses no condensing action. Neither potash, soda, or the hydrates of the oxides of zinc, copper, and cadmium react on formol; with lime (30 per cent of the weight of the aldehyde), only 20 per cent of the aldehyde is transformed into formose, while with hydrated amorphous oxide of lead the return reaches 70 per cent.—*Rev. Trav. Chim. P.-B.*, xviii., p. 309.

Opoponax.—A. Knit.—The composition of this resin is as follows:—The first part, soluble in ether, comprises an ether of ferulaïc acid and of oporesinotannol; the second part, insoluble in ether, comprises the free oporesinotannol, a gum, an essence, free ferulaïc acid, vanilline, and bassorine. The oporesinotannol alcohol gives a monobenzoylised derivative answering to the formula $C_{12}H_{13}O_2(OH)$. The gum, which does not act on polarised light, corresponds to the formula $C_8H_{14}O_7$. The essential oil gives a crystalline body, $C_{20}H_{10}O_7$, fusible at 133—134°, soluble in warm water, and having an acid reaction. Opoponax is characterised by this double fact:—(1). That it contains a resinotannol, the molecule of which contains 6 atoms of C of the same centesimal composition as that of Siamese benzoin; and (2) that it belongs to the group of resins which do not give ombelliferone by distillation. As a complement to this research the author has also analysed galbanum, but without having been able to isolate the galbanic acid described by Hirschsohn. As galipot readily gives this acid, the author is of the opinion that galbanum may contain this resin in the form of an impurity. Galbanum, the same as sumbul root, gave ombelliferone on analysis. —*Arch. d. Pharm.*, ccxxxvii., p. 256.

The Action of Nitric Acid on the Saturated Carbides.—V. Markovnikof.—In previous papers the author has shown that concentrated HNO_3 acts very slowly at the ordinary temperature on the normal paraffins, but that it reacts easily on the saturated hydrocarbides containing a tertiary H. His research on trimethylethylmethane has shown that concentrated HNO_3 also only reacts very slowly in the cold on the carbides of the type R_4C, where R represents CH_3 or a normal radical $CH_3(CH_2)^n$; to all appearance the reaction becomes a little easier as the chain $CH_3(CH_2)^n$ lengthens, but it never becomes rapid. On penta-, hexa-, and heptamethylene, HNO_3, or a mixture of HNO_3 and H_2SO_4, only reacts at 100°; but with the substituted polymethylenes (containing tertiary H) nitric acid alone behaves as with the paraffins containing tertiary H; the sulphonitric mixture, on the contrary, requires a slight elevation of temperature. Continuing, the author gives a practical method for detecting the presence of carbides containing tertiary H in a mixture of saturated carbides, and for effecting quantitative estimations.—*Journ. Soc. Phys. Chem. R.*, vol. xxxi., p. 530.

The Action of Acetylene on Copper.—H. Alexander. —Acetylene has no action in the cold on pure precipitated copper; but when the temperature is kept at about 240° to 250°, the gas is absorbed with the formation of small quantities of a greenish liquid, having the smell of a hydrocarbide, and a solid body of a bright brown colour, similar to cork in appearance, which gradually fills and finally obtrudes from the back end of the tube. No trace of acetylene has been obtained from this substance which is not soluble in any reagent. When heated in air, it gives off yellowish fumes, smelling of Stockholm tar, and then takes fire and burns with brilliant incandescence. It contains about 2 per cent of copper, which, however, can be eliminated by means of hydrochloric acid and ferric chloride, without any alteration in the properties of the substance. Further experiments are necessary to determine the constitution of this body. Provisionally the author recognises that the copper plays the *rôle* of contact agent, and effects the molecular condensation of the acetylene in the form of this hydrocarbide resembling cork. The hydrogen resulting from this condensation does not escape in the free state, but helps in the formation of the liquid smelling of petroleum, which has been mentioned above.—*Berichte*, xxxii., p. 2381.

NOTES AND QUERIES.

*** Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Indiarubber.—Can you inform me of a good book treating the subject of quantitative analysis of manufactured indiarubber?—W. A. W.

MEETINGS FOR THE WEEK.

Wednesday, 20th.—Microscopical, 8. Demonstration on the Structure of some Palæozoic Plants, with Sections of the Plants thrown on the Screen, by William Carruthers, F.R.S., President.

—— Institution of Mining and Metallurgy, 8. "On the Extraction of Gold from Auriferous Pyrites by Amalgamation," by T. G. Davey. "Notes on Gold-bearing Gravels," by W. S. Welton. "On the Treatment of Kalgoorlie Sulpho-Telluride Ores," by Alfred James.

Thursday, 21st.—Chemical, 8. Ballot for the Election of Fellows. "Notes on the Chemistry of Chlorophyll," by L. Marchlewski, Ph.D., and C. A. Schunck. "Researches on Morphine—I." by S. B. Schryver, D.Sc., and F. H. Lees. "A New Series of Pentamethylene Derivatives—I.," by W. H. Perkin, jun., F.R.S., J. F. Thorpe, Ph.D., and C. W. Walker. "Experiments on the Synthesis of Camphoric Acid—III. The Action of Sodium and Methyl Iodide on Ethyl-dimethylbutane Tricarboxylate, by W. H. Perkin, jun., F.R.S., and J. F. Thorpe, Ph.D. "On the Oxime of Mesoxamide and some Allied Compounds," by Miss M. A. Whiteley, B.Sc. "The Oxyphenoxy- and Phenyleneoxy-acetic Acids," by W. Carter and W. T. Lawrence, B.A., &c. "The Condensation of Ethyl-α-bromo-isobutyrate with Ethyl Malonates and Ethyl Cyanacetates: — α-Methyl-α'-isobutylglutaric Acid" and "Methylisoamylsuccinic Acid—II," by W. T. Lawrence, B.A., &c.

Friday, 22nd.—Physical, 8. (In the Rooms of the Chemical Society). "Notes on Gas Thermometry," by Dr. P. Chappuis. "A Comparison of Impure Platinum Thermometers," by H. M. Tory. "On the Law of Cailletet and Mathias and the Critical Density," by Prof. J. Young, F.R.S.

TO CORRESPONDENTS.

A. W. Comber.—Apply to the Assistant Secretary of the Chemical Society, Burlington House.

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Michaelmas Term.—Monday, October 1, to Saturday, December 15.

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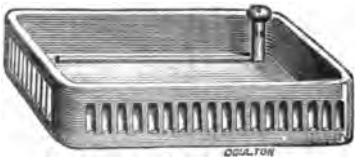

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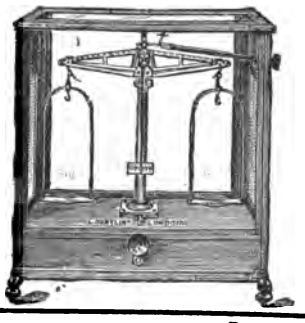

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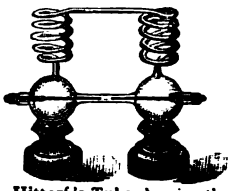

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Vol. LXXXI., No. 2117.

SOME NEW OBSERVATIONS ON THE STATIC DIFFUSION OF GASES AND LIQUIDS, AND THEIR SIGNIFICANCE IN CERTAIN NATURAL PROCESSES OCCURRING IN PLANTS.*

By HORACE T. BROWN, F.R.S., LL.D., and F. ESCOMBE, B.Sc., F.L.S.

This paper is intended to be the first of a series descriptive of the work carried out by the authors in the Jodrell Laboratory on the fixation of carbon by green plants, and deals mainly with the purely physical processes by which atmospheric carbon dioxide gains access to the active centres of assimilation.

The new evidence which F. F. Blackman brought forward in 1895 in favour of the gaseous exchanges of leaves taking place exclusively through the stomatic openings, presents at first sight certain difficulties of a physical nature, which have led to an examination of the whole question of the free diffusion of carbon dioxide at very low tension, and under a set of conditions very different from those under which the previous determinations of the coefficient of diffusion of carbon dioxide and air have been made by Loschmidt and others, where the gases were initially of equal tension, and the ratios of mixture departed widely from those of ordinary atmospheric air. The inquiry has led to the discovery of some new facts connected with the static diffusion of gases and liquids, which are of considerable interest, not only from the physical point of view, but from the explanations they suggest of certain natural processes which are primarily dependent on diffusivity.

The method employed in the first instance for the determination of the diffusivity of atmospheric carbon dioxide was one of *static diffusion* down a column of air of a definite length, towards an absorptive surface at the bottom of the column. When a static condition has been established, there is a steady flux of the carbon dioxide down the air column which may be quantitatively investigated by the same simple mathematical treatment as the "flow" of heat in a bar when the permanent state has been reached, or the "flow" of electricity between any two regions of a conductor maintained at a constant difference of potential.

By a long series of experiments of this nature it was found that the diffusivity constant, k, for very dilute CO_2 does not materially depart from the value assigned to it by Loschmidt and others, when experimenting with much higher ratios of mixture, and that the difference is certainly not of sufficient magnitude to be taken into serious account in the study of the natural processes of gaseous exchange in the assimilating organs of plants.

In the static diffusion of a gas, vapour, or solute, as the case may be, the amount of substance diffusing in a given time, all other conditions being the same, is directly proportional to the sectional area of the column. It is found, however, that if the flow is partially obstructed by interposing at any point in the line of flow a thin septum pierced with a circular aperture, the rate of flow across unit area of the aperture is greater than it would be across an equal area of the unobstructed cross-section of the column at this point. If the margin around the aperture has a width of at least three or four times its diameter, the rate of flow is now found to be directly proportional to the *linear dimensions* of the aperture and not to its area, so that the velocity of flow through unit area varies inversely as the diameter.

A large number of experiments on the diffusion of carbon dioxide, water-vapour, and sodium chloride in solution, are given in support of this proposition. All these show that the rate of diffusion across such a septum, all other conditions being the same, is directly proportional to the diameter of the aperture, and not, as might have been expected, to its area.

Exactly the same result is obtained when small circular discs of an absorbent, such as a solution of caustic alkali, are surrounded by a wide rim and exposed to *perfectly still* air, the amount of carbon dioxide absorbed under these conditions being proportional to the *diameters* of the discs.

If, however, there are any sensible air currents the absorption becomes proportional to the areas.

These two sets of phenomena may be explained as follows:—

In the case of the absorbing disc in perfectly still air, the convergent streams of carbon dioxide creep through the air towards the absorbing disc, establishing a steady gradient of density, and this creep will be a flux perpendicular to the lines of equal density, which form curved surfaces or "shells" surrounding the disc and terminating in the rim. The state of things is exactly analogous to the electric field in the neighbourhood of a conductor of the same shape and dimensions as the absorbent disc.* In the case of the gas, the curves or "shells" of equal density are the analogues of the similarly curved surfaces of equipotential above the electrified disc, whilst the converging lines of creep or flux of the gas are the analogues of the lines or tubes of force which bend round into the disc as they approach it.

If we consider two such absorbent discs of different diameters, the curved surfaces in each system corresponding to a given density will be found at actual distances from the discs, which are in the same proportion to each other as are the diameters of the discs. In other words, the gradient of density on which the rate of flow depends will be proportional to the diameters of the discs, which is exactly what is found experimentally.

This case of an absorbent disc is the exact converse of one which has been theoretically investigated by Stefan, viz., the conditions of evaporation of a liquid from a circular surface. He found that the lines of flux of the vapour proceeding from the surface of the liquid must be hyperbolas, whilst the curved surfaces of equal pressure of the vapour must form an orthogonal system of ellipsoids, having their foci, like the hyperbolas, in the bounding edges of the disc. This was a purely mathematical deduction which has never been verified experimentally, but it will be seen that the exactly converse phenomena of diffusion are in complete agreement with it.

In the other case of a diffusive flow through a circular aperture in a diaphragm, the lines of flow, which are *convergent* as they approach the aperture, bend round their foci situated in the edges of the disc and form a *divergent* system on the other side. If the chamber into which they pass is a perfectly absorbent one, and is sufficiently large, there will be formed on the inner side of the diaphragm a system of density shells similar to those outside, but with the gradient of density centrifugally instead of centripetally arranged. This system of shells is termed negative, and is as effective as the outer positive system in regulating the flow according to the "diameter law," so that this law will still hold good, even if the outer air currents are sufficient to sweep away the external positive shells altogether.

All the known facts of diffusion through circular apertures in a diaphragm are in complete accord with the above explanation, which is fully elaborated in the original paper.

* Abstract of a Paper read before the Royal Society, June 14, 1900

* The authors are indebted to Dr. Larmor for this suggestion of the electrostatic analogy.

By diffusing colouring matter through apertures in a septum, under such conditions as to prevent convection currents, the "density shells" have been rendered visible, and it has been shown that their ellipsoidal form is exactly that which is demanded by the above hypothesis. Moreover, this method gives an experimental demonstration of the more rapid projection of the diffusing particles from the edges of the aperture than from a point nearer its centre, a fact completely in harmony with the deduction of Stefan regarding the evaporation of liquids under analogous conditions.

The various cases which present themselves in practice with regard to the rate of diffusion through single apertures in a diaphragm are then discussed from the above point of view, and simple formulæ for the determination of this rate for single and double systems of density shells are established: (1) for cases where the thickness of the diaphragm is negligible, and (2) for other cases where the apertures become more or less tubular. In a subsequent section of the paper it is shown how closely the observed facts conform to these deductions, and that in static diffusion through apertures in a septum we have a new and accurate method for the determination of the diffusivity constants of atmospheric CO_2, of the vapours of liquids, and of substances in a state of solution.

Since the velocity of the diffusive flow through unit area of an aperture in a diaphragm varies inversely with the diameter, it might reasonably be expected that a diaphragm could be so perforated with a series of very small holes arranged at suitable distances from each other, as to exercise little or no sensible obstruction when it was interposed in a line of diffusive flow, although the aggregate area of the small holes might represent only a small fraction of the total area of the septum. Multiperforate diaphragms of this kind were found to possess all the remarkable properties which had been anticipated.

The material used for the septa was very thin celluloid, which was perforated at regular intervals with holes of about 0·38 m.m. in diameter. Details of a number of experiments with such diaphragms are given, in which it is shown that they may be so arranged as to produce but little obstructive influence on the diffusive flow of a gas when the total area of the apertures amounts only to about 10 per cent of the area of the septum, and that nearly 40 per cent of the full diffusive flow may be maintained when the number of the apertures is so far reduced as to represent an area of only 1·25 per cent of the full area of the septum.

The explanation is to be found in the local intensification of the gradient of density in the immediate neighbourhood of the diaphragm, and which does not extend to the column away from the apertures. This disturbance of gradient is brought about by the rapid convergence of the lines of flux, and their divergence on the other side, with the consequent formation of a system of "density shells" over each aperture. A system of perforations of this kind may be compared with a system of conductors electrified to a common potential, the density of the diffusing substance above the apertures corresponding to electric potential, and the non-absorbing portions of the diaphragm to a surface formed by lines of electric force. Just as the electric capacity of a plate is not much reduced by cutting most of it away, so also is it possible to block out a large portion of the cross section of the diffusing column without materially altering the general static conditions on which the flow depends.

The importance of these results in relation to diffusion through porous septa is next considered, diffusion through a thin porous septum being only an extreme case of free diffusion through a multiperforate diaphragm, whose apertures are so far reduced in size as to materially interfere with the mass movement of the diffusing substance.

A section of the paper is devoted to the application of these new observations to the processes of gaseous and liquid diffusion in living plants, and it is pointed out that the structure of a typical herbaceous leaf illustrates in a striking manner all the physical properties of a multiperforate septum. Regarded from this point of view it is shown that the stomatic openings and their adjuncts constitute even a more perfect piece of mechanism than is required for the supply of carbon dioxide for the physiological needs of the plant, and instead of expressing surprise at the comparatively large amount of the gas which an assimilating leaf can take in from the air, we must in future rather wonder that the intake is not greater than it actually is.

From data afforded by actual measurements of the various parts of the stomatal apparatus of the sunflower, it is shown that an extremely small difference of tension of the carbon dioxide within the leaf, as compared with that in the outer air, will produce a gradient sufficient to account for the observed intake during the most active assimilation.

It is also shown that the large amounts of water-vapour which pass out of the leaf by transpiration are well within the limits of diffusion, and that it is unnecessary to assume anything like mass movement in the outcoming vapour.

The translocation of solid material from cell to cell in the living plant is next considered, especially with reference to this transference, being, at any rate in part, brought about by means of the minute openings in the cell-walls through which the connecting threads of protoplasm pass. Notwithstanding the very small relative sectional area of these perforations they probably exercise an important function in cell-to-cell diffusion, in virtue of their properties as multiperforate septa.

There are two appendices to the paper, one in which a full description is given of a series of experiments on the absorption of carbon dioxide by solutions of caustic alkali from air in movement; the second being devoted to a detailed description of the methods used for accurately determining the carbon dioxide absorbed.

PREPARATION OF AN AMMONIACAL SOLUTION OF CUPROUS CHLORIDE, BY MEANS OF HYDROXYLAMINE, FOR THE DETECTION OF ACETYLENE.

By L. ILOSVAY von N. ILOSVAY.

HYDROXYLAMINE is a very convenient substance to use for the reduction of cupric chloride to cuprous chloride; but the colour of the precipitate produced by acetylene in a similar solution varies considerably with the proportions of the constituents.

The author proposes to prepare the solutions necessary for the detection of acetylene by means of the three following formulæ:—

1. Cupric chloride ($CuCl_2,3H_2O$) .. 0·75 grm.
   Chloride of ammonium .. .. .. 1·5 grms.
   Chlorhydrate of hydroxylamine .. 2·5 ,,
   Ammonia (20 per cent NH_3) .. .. 3 c.c.
2. Cupric nitrate [$Cu(NO_3)_2,5H_2O$] .. 1 grm.
   Hydrochlorate of hydroxylamine .. 3 grms.
   Ammonia (as above) .. .. .. 4 c.c.
3. Like No. 2, but the cupric nitrate is replaced by crystallised sulphate of copper.

The copper salt is dissolved in a little water in a 50 c.c. flask; the ammonia is added drop by drop, then the salt of hydroxylamine; shake carefully and make up the volume to 50 c.c. In a few minutes the solution becomes colourless and is then ready for use. It will keep well for two or three days, but at the end of this time its sensibility gradually diminishes.

Acetylene is tested for in illuminating gas by shaking up a few cubic centimetres of this solution with half a litre

of gas, or by passing the gas through a tube containing wadding or glass-wool impregnated with the reagent. The nitrate solution is the most sensitive, but the precipitate formed seems to be less stable than that obtained with the other solutions.—*Berichte*, xxxii., p. 2697.

A NEW METHOD FOR THE ESTIMATION OF MORPHINE IN OPIUM.

By H. M. GORDIN and A. B. PRESCOTT.

All the alkaloids of opium may be set free by ammonia. After a sufficient maceration the liquid is evaporated, and the narcotine, codein, thebain, papaverine, and most of the other bases soluble in C_6H_6 are removed by means of this solvent. The morphine, which is insoluble in C_6H_6, is removed by a mixture of $CHCl_3$ and C_2H_6O. The solvent is evaporated, the residue dissolved in a solution of 1/20th normal acid, and the excess of acid titrated by a solution of 1/20th normal KOH. This method can be checked by a titration with iodine on the product of the above estimation. It suffices to treat it with $Ca(OH)_2$, acidulate, and add a solution of titrated iodine; one molecule of morphine fixes 3 atoms of iodine, giving the compound $C_{17}H_{19}NO_3HI.I_3$. The iodine in excess can be estimated with $S_2O_3Na_2$. Both methods give identical results.—*Archiv d. Pharm.*, ccxxxvii., p. 380.

ON SELENIFEROUS SULPHURIC ACID.

By MM. SCHLAGDENHAUFFEN and PAGEL.

While preparing pure hydrochloric acid for laboratory use, by means of decrepitated or fused chloride of sodium and sulphuric acid, labelled pure as in the usual manner, we were astonished to find that the water in the wash-bottle became coloured red. The tint became deeper and deeper as the operation continued, and at the end of a certain time quite a noticeable deposit was produced. This latter soon disappeared, and the liquid again became colourless. If we then passed a current of sulphurous acid through the solution, a brick-red precipitate was again formed, analogous to the one first observed.

This deposit is due to nothing else but selenium. It can only come from the chloride of sodium or the sulphuric acid. Now, as the solution of the salt used did not undergo any change under the influence of a current of sulphurous acid, it is beyond doubt that the presence of the selenium could only be due to an impurity in the sulphuric acid.

Different samples of sulphuric acid, obtained from the makers at Nancy, having given us the same results, we looked about for other samples with the object of repeating the experiments. We were surprised to find that out of ten samples, labelled both pure and ordinary, there were only three which did not give first the red colour, and then a more or less abundant deposit after being heated for some time.

All the commercial acids examined during this period thus contained selenium, and the commercial processes now in use for getting rid of the arsenic are shown to be insufficient for removing the selenium at the same time.

One sample only, prepared by ourselves in our own laboratory, and rectified over bichromate of potassium, did not give this reaction, and was thus free from the impurity.

To detect this metalloid in sulphuric acid, it is not necessary to operate on a large quantity of acid at a time; a few drops are sufficient; in fact, as selenious acid, treated with concentrated sulphuric acid, has the property of colouring codein, first an intense green, then blue, we thought that this very characteristic reaction might be utilised in this particular case, the more so as pure sulphuric acid alone does not give the colouration with codein.

Thus, if we pour 5 or 6 drops of sulphuric acid over a fragment of codein, and if, after the lapse of a few minutes, there appears a green colouration in the cold, changing to a greenish blue on the water-bath, we may be certain that selenium is present in the acid under examination.

This sensitive reaction is of the same kind as that long known for detecting the presence of nitrated products in sulphuric acid, by means of the rose-colour obtained by placing the acid in contact with a fragment of brucine.—*Journ. Pharm. et Chim.*, Series 6, vol. xi., No. 6.

THE PURIFICATION OF ACETYLENE BY MEANS OF ACID SOLUTIONS OF CHROMIC ACID.

Doubts having been thrown on the efficacy of Ullmann's method of purifying acetylene by means of an acid solution of chromic acid, the inventor has published the results of some experiments carried out by Dr. Wachs, of Carlsruhe, which prove the efficacy.

Two glass tubes, 40 c.m. in height, were filled with pumice-stone impregnated with a solution obtained by dissolving 1 part of crystallised chromic acid in 2 parts of acetic acid at 50 per cent. Through these tubes was passed a quantity of acetylene containing 0·5066 grm. of phosphorus per cubic metre; the gas was then passed through a tube of smaller dimensions filled with lime and sawdust to absorb all traces of acetic acid carried over by vapourisation. The gas thus purified did not contain a trace of phosphorus when the rate of flow was 29 litres per hour.

At a speed of 119 litres per hour, which is much higher than that advised by the inventor, the gas on leaving contained a small quantity of phosphorus.

The lighting power of the gas was determined before and after purification; it was practically the same.

It is stated that 5·5 grms. of chromic acid suffice to purify 1 cubic metre of acetylene, and that the cost of purification by this method represents 0·3 per cent of the cost of the carbide used for the production of the gas.

It has also been stated that Ullmann's method resembles that of Frank for the purification of acetylene. Ullmann, on the contrary, says that the acidulated solutions of the metallic salts, as used by Frank, retain the impurities, owing to the formation of insoluble or non-volatile compounds with the metals. Chromic acid acts quite differently; it oxidises the impurities instead of forming precipitates with them.

It has further been stated that in Ullmann's process the active body is not chromic acid, but a solution of chromic chromate. According to Ullmann, however, such a solution cannot exist, for, even if it were formed, it would immediately decompose into active chromic acid and an inactive chromic salt. Further, experiments have shown that an acidulated solution of chrome-alum will not completely remove the impurities from acetylene.

It may be pointed out that the estimation of the expense of the purification of acetylene by the various methods is not correct, as they do not give the analysis of the raw gas or its rate of flow, and, further, the purity of the gas obtained has not been proved by trustworthy experiments.

As reagents for determining the degree of purity of acetylene, Ullmann recommends:—1. An aqueous or slightly acid solution of acetate of lead at 10 per cent; 2. A solution of 1 part of bichloride of mercury in 10 parts of hydrochloric acid at 10 per cent.

The gas is passed through two wash-bottles containing respectively these two solutions. If it has been imperfectly purified, white rings form on the tube through which the gas comes, and finally the solutions become cloudy.—*Journ. f. Gasbeleuchtung*, xlii., [12], p. 198.

INFLUENCE OF FINELY-DIVIDED PLATINUM ON THE COMBINATION OF HYDROGEN AND OXYGEN.

By WM. FRENCH, M.A., F.I.C.

It has been long known that many mixtures of gaseous bodies can be readily made to combine chemically if they be in contact with spongy platinum (*e.g.*, a mixture of hydrogen and oxygen at ordinary temperatures, or hydrogen and bromine vapour, &c.); but, so far as I am aware, no work has been published previous to my preliminary experiments (*Proc. Chem. Soc.*, 1897, No. 175, p. 52) on the action of this so called catalytic agent in presence of highly purified gaseous bodies, *i.e.*, gases in which all traces of moisture or other electrolyte have been removed.

The following work was therefore undertaken with a view of ascertaining, if possible, the part played by platinum in these changes. Most of the experiments were completed in 1897; since then, however, I have confirmed them, the whole of which are summed up below.

My first experiments consisted of allowing purified bromine vapour and hydrogen gas to come in contact with spongy platinum. The difficulties experienced were so many that I abandoned them *pro tem.* after the preliminary trials. The results obtained, however, were sufficiently satisfactory to induce me to continue the work, using the gases hydrogen and oxygen.

Briefly, these experiments consisted in allowing carefully dried hydrogen and oxygen gases (prepared electrolytically by a Bunsen decomposer) to come into contact with carefully purified and dried spongy platinum, both in presence and in absence of light.

Details of the Work.

Preparation of Spongy Platinum.—This was obtained from foil by solution in aqua regia, precipitation as double chloride with ammonium chloride, and careful ignition at as low a temperature as possible. This was well washed and dried *in vacuo* in contact with a tube of fused caustic potash.

Drying Reagents Used:—

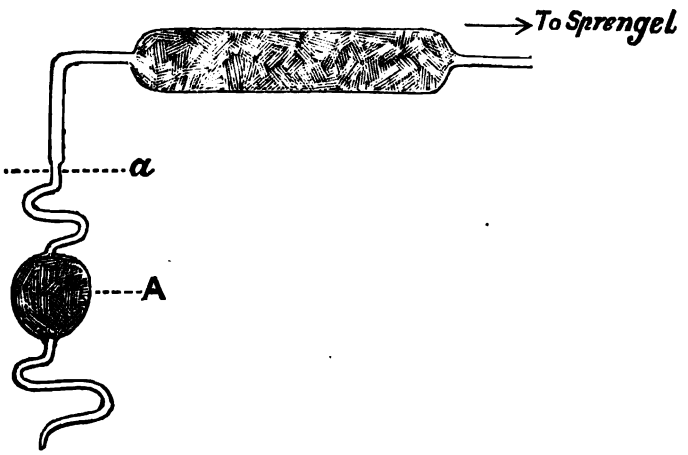
1. *Re-distilled sulphuric acid.*
2. *Potash.*—Prepared by alcohol from Messrs. Hopkin and Williams, and fused as suggested by Dittmar and Henderson (Chemical News, lxiii.).
3. *Phosphoric Anhydride.*—In some of my first experiments this was purified as suggested by Shenstone (*Journ. Chem. Soc.*, lxiii.), but later on I used the purest, obtained from Messrs. Hopkin and Williams.

Charging the Drying Tubes.—Some of the potash drying tubes were connected, after charging, with a phosphoric oxide tube, which was connected to an exhaust pump. The combination was exhausted, and then air allowed to quickly enter, so that in passing over the phosphoric oxide it carried small particles forward, which became scattered on the fused potash; this I found made a very efficient drying tube. Some of the phosphoric oxide drying tubes were filled in the following way:—Some glass-wool was chopped as finely as possible with a knife, then placed in a wide-mouthed stoppered bottle, and heated in an air-bath to about 300° C., and cooled in a desiccator. After cooling until the bottle was just warm, about an equal volume of phosphoric oxide was added and the two thoroughly shaken together. By this method the quantity of oxide required was very greatly diminished, whilst the absorbing surface was largely increased. On some occasions, on using a tube containing this mixture, I noticed a smell of phosphine, and so abandoned this form of drying tube in my later experiments.

Description of Apparatus.

1. About 3 to 4 grms. of spongy platinum for each experiment were placed in a bulb of about 5 c.c. capacity and drawn out and bent as in A, Fig. 1. This was connected to a phosphoric oxide tube and Sprengel pump.

FIG. 1



The bulb was placed in an air oven and heated for eight to twelve hours at a temperature of about 200° C. *in vacuo*, then sealed off at *a*, Fig. 1. The object of the bent drawn out ends was to insure rupture of the bulb, and consequent exposure of the platinum with the gases when necessary.

2. The sealed bulb containing the platinum, A, was next placed in a tube, B (Fig. 2), between two constrictions, *b b'*. This tube was about 50 c.m. long between the constrictions and 2 c.m. diameter. At each end of B, beyond the constrictions, was a *well ground* stopcock; beyond the stopcocks were drying tubes (C, D, E); C and D contained phosphoric oxide, whilst E contained caustic potash, and each tube was about 25 c.m. by 2 c.m.; F and G contained re-distilled sulphuric acid; G fitted into the decomposer, H, by a ground joint, I, and mercury trap, J. The end of the drying tube, C, was drawn out and bent, *c''*, so that it could be used as a delivery tube or connected to an exhaust pump. The whole apparatus had *fused glass joints*, and was about 200 c.m. long.

Experimental.

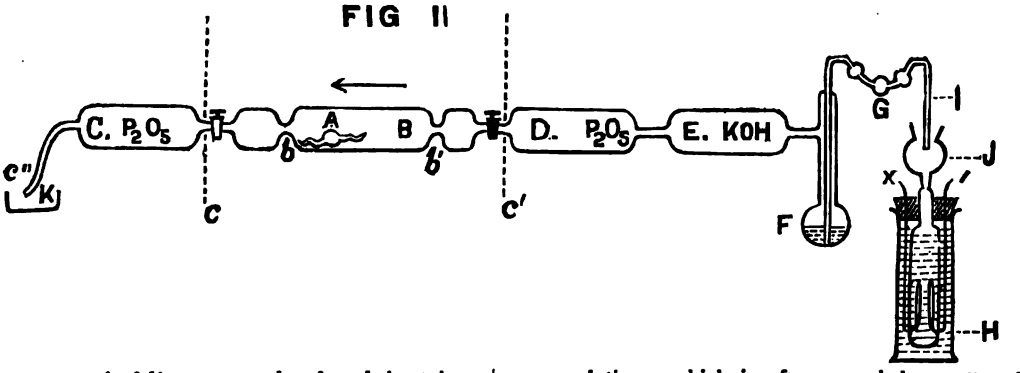
1. The tube *c'* was connected to a Sprengel's pump, and the ground-glass joint I, after removing from the mercury trap J, was attached to two or three wash-bottles, a current of air was then slowly drawn through the whole

apparatus for five to eight hours, whilst the whole of the tube B was heated to near the softening-point of glass; this was to ensure the removal of any gases, &c., adhering to the sides of the tube.

2. The joint I was now replaced in the mercury trap J, and a current from a three-celled battery passed through the decomposer for about two hours; the delivery tube *c''* was then placed beneath mercury in a trough, K, and the electrolytic gases passed for about eight to twelve hours.

Note.—Tubes of the escaping gases were collected from time to time, and small quantities of spongy platinum added (this had not been previously dried as described above). In all cases the gases combined (if sufficient platinum were added) with an explosion, and in no case did I use more platinum than was actually used in the experiments, viz., 3 to 4 grms. But I noticed in all cases a certain interval of time to elapse between the addition of the platinum and the actual explosion. This interval seemed to me to be less in broad daylight, or when burning a piece of magnesium near to the tube, than at night when most of the experiments were carried out.

3. The whole apparatus, now filled with the electrolytic gases, was wrapped in black paper and allowed to stand for periods varying from three to six days (later on, in one experiment, I found one day sufficient to dry the gases); the stopcocks *b b'* remained open, so that the gases were in direct contact with the drying reagents.



4. The stopcocks *b b'* were now closed, and the tube B separated from C and D by a file at *c c'*. B (being still wrapped in black paper) was shaken, so that the bulb A containing the platinum was smashed. In *no case* did an explosion occur, nor was there any appearance of a formation of dew, or heat evolution, even after a lapse of fifteen to twenty minutes.

5. The tube B was next divested of its wrapper and exposed to bright sunlight when available, or a strip of burning magnesium ribbon; still, in *no case* was there any appearance of chemical combination.

6. The tube B was then opened and a lighted match applied, and in all cases the mixture exploded.

These experiments, only differing slightly in detail, having been repeated so often, I was led to the conclusion that even if spongy platinum does bring about the combination of these gases by itself at ordinary temperatures, then the combination must be extremely slow, and, for an actual explosion to take place, a third body must also be present.

I next tried to find the conditions necessary for the gases to rapidly combine (*i. e.*, explode); so far I have only tried the addition of one substance, *viz.*, water, and even the results from this are not satisfactory.

The following gives a general result of the work in this direction:—

7. The tube B, still wrapped in paper, and containing the gases and platinum *in contact*, as obtained in 4, was cooled in a tube of water, and a plug of cotton-wool, damped with water, placed above one of the openings *c c'*; the stopcock next to the plug was quickly opened and closed, so that an inrush of damp air was admitted; but in *no case* did I notice any sign of combination, even after fifteen minutes. In some cases more moist air was introduced, and in one or two cases a drop of water (distilled) added, yet no explosion was observed.

8. The tube was again divested of its paper cover, and exposed to strong light, as before; and only on one occasion* did I get any trace of an explosion, or sign of combination. In every case I now opened the tube, and applied a lighted match, and at once obtained a flash, showing that, even if combination had taken place at all, it must have been extremely slow.

Many circumstances have prevented me continuing the work, although I have made preliminary trials, using the gases NO_2 and H in presence of platinum, also HCl and O in presence of copper sulphate and pumice. Should I get the opportunity I intend continuing the work.

Conclusions.

1. Finely-divided platinum does not, by itself, bring about the combination of a mixture of purified hydrogen and oxygen at ordinary temperatures.

2. The combination of these gases, when not carefully dried, in presence of spongy platinum seems to be influenced by light.

It seems, then, that the so-called "catalytic action" of spongy platinum, which is often regarded as a "surface action," may probably only be true when in presence of an electrolyte, as is possibly the case with many other mixtures of gaseous bodies, which, although they readily combine under ordinary circumstances, will not do so if quite pure and dried, as shown by Dixon, Baker, and others.

Since conducting the above experiments, which were all, with one exception, made previous to 1899, seeing that the action was evidently not by itself a truly surface action,—*i. e.*, the energy necessary to bring about the change was not at any rate *all* derived from the condensation of the gases on the surface of the platinum,—I was led to roughly calculate the heat which would be generated, and the temperature to which the mixture of gases and platinum might be raised by such condensation. This I find is lower than 612° C., which is given by Meyer as the point of ignition of the electrolytic gases.

Neumann says platinum sponge absorbs about fifty times (49·3) its own volume of hydrogen gas.

Now the greatest mass of platinum taken was never greater than 5 grms., and taking 20 as the sp. gr. of platinum, this would give 0·25 c.c. as the greatest volume of platinum taken for any individual experiment; therefore the volume of hydrogen condensed would not be greater than about 12 c.c. Then, taking sp. ht. of platinum as 0·032, and that for hydrogen as 3·4, the increase

* In this particular case I fancied I had a trace of HCl on the cotton-wool plug.

in temperature of the mass (neglecting oxygen) is below 600° C., which is below the point of ignition of the mixture.

Grammar School, Bury.

PROCEEDINGS OF SOCIETIES.

THE ROYAL SOCIETY CONVERSAZIONE.

On Wednesday evening (June 20th) the second Conversazione, known as "the Ladies' Night," was held at the Royal Society's Rooms at Burlington House, the Fellows and guests being received by the President, The Lord Lister, F.R.C.S., D.C.L.

Among the numerous and interesting exhibits we may mention that of Prof. Ramsay and Dr. Travers, who showed the following:—

(1). The Inert Atmospheric Gases; their Spectra, and some of the Apparatus Used in Determining their Physical Properties.

Spectrum of—	Density.	Atomic weight.
Helium	1·98	3·96
Neon	about 10	about 20·0
Argon	19·96	39·92
Krypton	40·77	80·54
Xenon	64·0	128·0

(2). Apparatus for Determining the Molecular Volume of these Elements in the Liquid State.

(3). Apparatus for Distilling the Gases Fractionally; Fractionation of Atmospheric Air.

(4). Bulbs in which the Densities of the Gases have been Determined.

Mr. J. Mackenzie Davidson, M.B., exhibited a Stereoscopic Fluoroscope, by means of which an observer is enabled to see the shadows cast by Röntgen rays on the fluorescent screen in stereoscopic relief. With this instrument, a bullet or needle, &c., can be seen in its true position and its anatomical relations observed, and the surgeon can easily touch the bullet, &c., with a probe or forceps, while he is looking at its shadow on the screen; the position of a Mauser bullet in a leg of mutton was distinctly found by this means.

The same gentleman also showed a Rotatory Mercury Break, with which good results are obtained with from 12 volts up to 100, according to the speed of rotation.

The Eclipse Committee of the British Astronomical Association showed a number of interesting Drawings and Photographs of the Total Eclipse of the Sun on the 28th of May last.

A further number of Photographs and Drawings of the same subject were also exhibited by the Royal Astronomical Society and the Joint Permanent Eclipse Committee of the Royal and Royal Astronomical Societies.

Prof. Silvanus Thompson, D.Sc., showed some experiments on the Aberration called *Coma*. Coma is an aberration due to the several zones of the lens not having equal focal lengths, and hence, when the lens is transmitting an oblique pencil, the unequal refraction of the different parts gives rise to a singular unilateral distortion of the cones of rays traversing the various zones. The actual path of the rays was very distinctly shown by means of a smoke box which they traversed.

Mr. W. Gowland exhibited some Remains of a Roman Silver Refinery found at Silchester. The hearths, which are now saturated with lead and copper oxides, were constructed of bone-ash; they are of special interest, being the only examples of Roman cupellation furnaces of that material which have been found.

Professor Hele-Shaw and Mr. Hay exhibited at the May Soirée of last year some experiments in stream line motion which were analogous to lines of magnetic induction. These were produced by the method of thin films of variable thickness, the greater depth corresponding to greater permeability of the analogous magnetic body. Since that period the accurate measurement and calculations necessary for determining to what degree these results corresponded to the known laws of magnetic induction have been carried on. A paper read before the Royal Society, on Thursday, the 21st inst., gives a full account of this work, and also of the improvements in the actual appliances used, which latter may be stated briefly to be—

1. The use of a translucent medium which can be accurately shaped and made of a required thickness with a perfectly true surface.
2. The adoption of a specially constructed glass plate by which a large number of results may be more readily obtained with comparatively little trouble, and which will enable the method to be generally adopted for educational work, as well as for purposes of research.

Prof. Ray Lankester showed some enlarged models of Gnats and of Human Blood Corpuscles Infected by the Malaria Parasite; these latter are magnified seven thousand five hundred times.

Three demonstrations, with experiments and lantern slides, were given in the Meeting-room, viz., Cinematograph Photographs of Native Dances in Torres Straits, by Prof. Haddon; the production of Short Electric Waves and the Study of Electro-optic Phenomena, by Prof. J. A. Fleming; and the Life-history of the *Cicindela campestris*, —the Common Tiger Beetle.

CHEMICAL SOCIETY.

Ordinary Meeting, June 7th, 1900.

Professor Thorpe, C.B., F.R.S., President, in the Chair.

Messrs. T. G. Joyce, A. W. Bain, T. FitzGibbon, and B. Jordan-Smith were formally admitted Fellows of the Society.

The following certificate was read for the first time:—Edmund Foster Hudson, Churcher's College, Petersfield, Hants.

Professor Dunstan, on behalf of the Society, congratulated the President on the distinction recently conferred upon him by Her Majesty.

The President returned his thanks to the Society for their congratulations.

Of the following papers, those marked * were read:—

*75. "*Condensation of Ethyl Acetylenedicarboxylate with Bases and δ-Ketonic Esters.*" By S. Ruhemann and H. E. Stapleton.

The esters previously obtained (*Trans.*, 1896, lxix., 530, 1383; 1897, lxxi., 323) by the action of ethyl acetoacetate and ethyl benzoylacetate on ethyl chlorofumarate had been formulated thus:—

$$CO_2Et\cdot C{:}CH\cdot CO_2Et \qquad CO_2Et\cdot C{=\!=}CH\cdot CO_2Et$$
$$\Big| \qquad \text{and} \qquad \Big| \qquad .$$
$$CO_2Et\cdot CH\cdot CO\cdot CH_3 \qquad CO_2Et\cdot CH\cdot CO\cdot C_6H_5$$

These esters were found by the authors to be identical with those which are formed by using ethyl acetylenedicarboxylate instead of ethyl chlorofumarate (*Trans.*, 1899, lxxv., 784). The considerations brought forward (*Trans.*, 1900, lxxvii., 242) led to the conclusion that these esters should be represented by the formulæ—

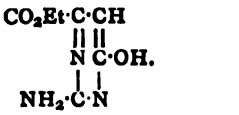
$$CO_2Et\cdot C\cdot CH_2\cdot CO_2Et \qquad CO_2Et\cdot C\cdot CH_2\cdot CO_2Et$$
$$\| \qquad \text{and} \qquad \| \qquad .$$
$$CO_2Et\cdot C\cdot CO\cdot CH_3 \qquad CO_2Et\cdot C\cdot CO\cdot C_6H_5$$

Ethyl acetoaconitate. Ethyl benzoylaconitate.

The latter expressions are in harmony with the facts

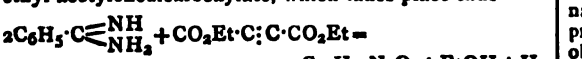
that these esters do not give a colouration with ferric chloride and that the same substances are produced by using chlorofumaric and chloromaleic esters (*Trans.*, 1896, lxix., 530).

The difference in the behaviour of ethyl acetylenedicarboxylate from that of ethyl phenylpropiolate is also found in its reactions with guanidine and benzamidine. The former base yields with it a cyclic compound which has both basic and acidic properties, and is regarded by the authors as ethyl amidohydroxypyrimidinecarboxylate,—

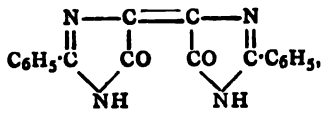


This ester, on boiling with potassium hydroxide, hardly undergoes any change beyond hydrolysis to the corresponding acid.

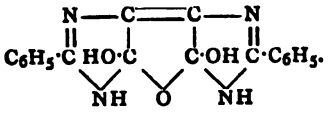
Quite unexpected is the reaction of benzamidine with ethyl acetylenedicarboxylate, which takes place thus—



(the hydrogen probably effects a reduction), producing ruby-red crystals of a substance called *glyoxaline red*. Its constitution is probably—



which indicates the close relationship of this compound to indigo and pyrazole blue. Glyoxaline red, when boiled with glacial acetic acid, takes up one molecule of water, and is transformed into a substance, $C_{18}H_{14}N_4O_3$, crystallising in yellow needles, for which the authors suggest the formula—

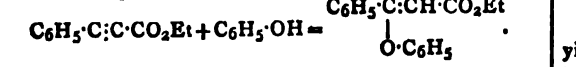


This compound dissolves in potassium hydroxide, forming a yellow solution, which gradually acquires the red-violet colour of the alkaline solution of glyoxaline red.

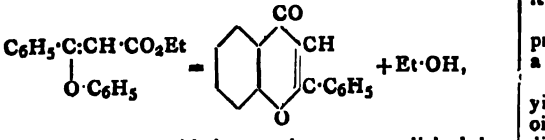
The authors have found that the same substances are formed from guanidine as well as from benzamidine by using ethyl chlorofumarate instead of ethyl acetylenedicarboxylate.

*76. "*Condensation of Phenols with Ethyl Phenyl propiolate.*" By S. Ruhemann and F. Beddow.

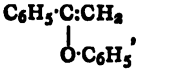
The ease with which the esters of the acetylene series form additive products induced the authors to study their behaviour towards phenols. They find that the sodium phenoxides readily combine with ethyl phenylpropiolate to form aryl derivatives of ethyl β-hydroxycinnamate; thus in the case of sodium phenoxide itself the following reaction takes place:—



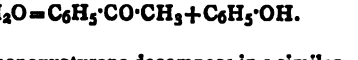
It was hoped that these esters might be converted into flavone and its derivatives, according to the equation—



but up to the present this has not been accomplished, because the esters are so easily hydrolysed to the corresponding acids, whilst the acids themselves, on heating, lose carbon dioxide and yield phenoxystyrene,—



and its homologues; further, on warming phenoxystyrene with dilute acids, it decomposes into acetophenone and phenol, thus—



The homologues of phenoxystyrene decompose in a similar manner.

The authors found that ethyl phenylpropiolate reacts with the sodium derivatives of *o*- and *p*-cresol to form compounds analogous to those obtained with sodium phenoxide. The α- and β-naphthols also react in a similar manner with the ester, and form the ethyl esters of the naphthoxycinnamic acids; thus from α-naphthol ethyl α-naphthoxycinnamate and the corresponding acid were prepared; this acid seems to be less stable than those obtained from phenol and the cresols, as it undergoes decomposition when left in contact with dilute sulphuric acid, acetophenone being produced.

*77. "*The Constitution of Pilocarpine.*" By H. A. D. Jowett, D.Sc.

In a recent paper (*Trans.*, 1900, lxxvii., 494), the author gave an account of some preliminary experiments on isopilocarpine, and reserved the question of the constitution of the alkaloid for a future paper. Owing to the publication of a paper on pilocarpine by Pinner and Kohlhammer (*Ber.*, 1900, xxxiii., 1424), the author has deemed it advisable to give a brief account of the results he has thus far obtained.

Action of Fused Potash on Isopilocarpine.—It was shown (*loc. cit.*) that ammonia, methylamine, and an acid, probably isobutyric, were formed in this reaction. It has now been proved that isobutyric acid is formed, in addition to other acids which are produced in quantity too small to be identified.

Distillation of Pilocarpine with Soda-lime.—A preliminary experiment had shown that the bases formed in this reaction are ammonia and either 2- or 3-methylpyridine. Further investigation has shown that, in addition, methylamine is formed, and that only a very small quantity of 3-methylpyridine is produced. As the formation of traces of a pyridine base by distillation with soda-lime does not throw very much light on the constitution of the alkaloid, this reaction has not been further investigated.

Oxidation of Isopilocarpine by Potassium Permanganate.—The oils previously mentioned have now been examined, and it has been proved that the products of oxidation, besides ammonia and methylamine, are acetic acid and an acid having the formula $C_7H_{10}O_4$. The quantity of permanganate required for the oxidation is about 6 molecular proportions, and the reaction may be expressed by the following equation:—

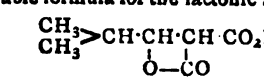
The new acid was obtained as the ethyl ester, and the yield was 70 per cent of the theoretical. The acid is a very slightly coloured yellow oil, with a peculiar smell, and boils at 210—220° at 10 m.m. pressure. By titration, it was shown to be a lactonic acid.

The ethyl ester, $C_9H_{14}O_4$, boils at 299° at atmospheric pressure, is optically active, $[\alpha]_D$ 15° = +30·76°, and has a specific gravity 1·1053 (15°/15°).

The ethyl ester reacts with phosphorus pentabromide, yielding the bromethyl ester, which was isolated as an oil boiling at 165—170° (20 m.m.). This was treated with diethylaniline, and the unsaturated ethyl ester, boiling at 155° (10 m.m.) hydrolysed, when an acid was obtained

which boiled at 180—200° (10 m.m.), and answered Baeyer's test for an unsaturated acid. It was now oxidised, first with cold permanganate, and then with chromic acid, to an acid which was proved by analysis of the crystalline silver salt to be isobutyric acid.

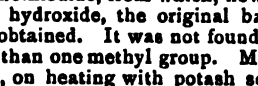
The probable formula for the lactonic acid is therefore—



Action of Methyl Iodide on Isopilocarpine.—When isopilocarpine methiodide is heated with potash solution in a sealed tube, the base formed is methylamine, thus proving that the nitrogen atom to which pilocarpine owes its basic character is the one eliminated in various reactions as ammonia. By treatment with silver hydroxide and subsequent heating, a new base is formed—*methylisopilocarpine*—which is insoluble in chloroform and forms a crystalline platinichloride, m. p. 218°, and a crystalline picrate, m. p. 136°. This base reacts with methyl iodide to form a methiodide, from which, however, by treatment with silver hydroxide, the original base—methylisopilocarpine is obtained. It was not found possible to introduce more than one methyl group. Methylisopilocarpine methiodide, on heating with potash solution in a sealed tube, yields only one base—methylamine. These experiments prove the existence of the group :NH in isopilocarpine, and, since the other nitrogen atom always yields methylamine, it must occur as the group :NCH_3. Herzig and Meyer have stated that one methyl group is attached to a nitrogen atom, and Chastaing was unable to prepare dialkyl derivatives.

Without proposing any formula for isopilocarpine, it may be desirable to state the facts which any formula for the base must explain:—

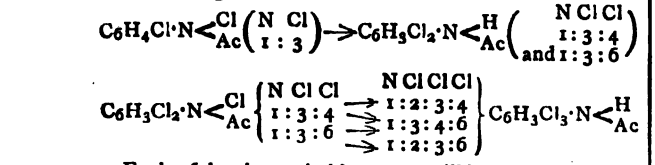
1. The optical behaviour of pilocarpine and isopilocarpine, and the remarkable stability of the latter base towards heat, alkalis, and various chemical reagents.
2. The non-basic character of the group :NCH_3.
3. The existence of the groups—



:NH, and :NCH_3.

*78. "*The Nitrogen Chlorides derivable from Meta-chloro-acetanilide and their Transformations.*" By F. D. Chattaway, K. J. P. Orton, and W. H. Hurtley.

The action of hypochlorous acid on meta-chloro-acetanilide was described. The nitrogen chlorides formed, and the transformations they undergo, are represented by the following scheme:—



Each of the three tri-chloro-acetanilides yields nitrogen chlorides, which change into the same tetra-chloro-acetanilide (NH : Cl : Cl : Cl : Cl = 1 : 2 : 3 : 4 : 6). In the transformation of these nitrogen chlorides in which one meta-position is occupied, the formation of the ortho-derivatives takes place to a greater extent than in the transformation of the nitrogen chlorides derived from acetanilide (*Proc.*, 1900, xvi., 112). The transformation is also a much slower and more difficult process.

The following substances are described:—

Metachlorophenylacetylnitrogen chloride (acetylchloramino-3-chlorobenzene), $C_6H_4Cl{\cdot}NCl{\cdot}COCH_3$. Clusters of small prisms, m. p. 93°.

3 : 4-*Dichlorophenylacetylnitrogen chloride* (acetylchloramino 3 : 4-dichlorobenzene), $C_6H_3Cl_2{\cdot}NCl{\cdot}COCH_3$. Clusters of very slender prisms, m. p. 92°.

2 : 5-*Dichlorophenylacetylnitrogen chloride* (acetylchloramino-2 : 5-dichlorobenzene), $C_6H_3Cl_2{\cdot}NCl{\cdot}COCH_3$. Small prisms, m. p. 73°.

2 : 3 : 4-*Trichlorophenylacetylnitrogen chloride* (acetylchloramino-2:3:4-trichlorobenzene), $C_6H_2Cl_3{\cdot}NCl{\cdot}COCH_3$. Clusters of flattened prisms, m. p. 113—114°.

2 : 4 : 5-*Trichlorophenylacetylnitrogen chloride* (acetylchloramino-2:4:5-trichlorobenzene), $C_6H_2Cl_3{\cdot}NCl{\cdot}COCH_3$. Rosettes of short, thick prisms, m. p. 96°.

2 : 3 : 6-*Trichlorophenylacetylnitrogen chloride* (acetylchloramino 2:3:6-trichlorobenzene), $C_6H_2Cl_3{\cdot}NCl{\cdot}COCH_3$. Small, four-sided prisms or plates, m. p. 116°.

2 : 3 : 4 : 6-*Tetrachlorophenylacetylnitrogen chloride* (acetylchloramino-2 : 3 : 4 : 6-tetrachlorobenzene), $C_6H_2Cl_4{\cdot}NCl{\cdot}COCH_3$. Clusters of small prisms, m.p. 97°.

*79. "*The Persulphuric Acids.*" By T. Martin Lowry, D.Sc., and John H. West.

This work was undertaken in order to elucidate the part which "persulphuric acid" plays in the accumulator (Armstrong and Robertson, *Proc. Roy. Soc.*, 1891, l., 105) and to ascertain to what extent the production of this acid must be taken into account in discussing the electric conductivity of solutions of sulphuric acid, as well as in determining the origin of the products which such solutions yield when electrolysed under various conditions (Armstrong, *Trans.*, 1895, lxvii., 1156).

In the course of the inquiry it became necessary to repeat and extend Berthelot's observations on the interaction of hydrogen peroxide and sulphuric acid (*Ann. Chim. Phys.*, 1878, [v.], xiv., 345—354). The experiments have led to results which appear to have an important bearing on the constitution of the product formed by the interaction of sulphuric acid and potassium persulphate which Caro (*Zeit. Ang. Ch.*, 1898, iv., 845) has shown to be possessed of special properties, and which Bamberger as well as v. Baeyer and Villiger have made use of as an characterised by Caro is a "persulphuric" acid composed oxidising agent under the name of "Caro's reagent." It has been suggested by v. Baeyer and Villiger that the acid of hydrogen peroxide and sulphuric anhydride in the ratio $H_2O_2 : SO_3$. The author's experiments favour the conclusion that its composition may be $H_2O_2 : 4SO_3$; the acid of which salts were first prepared by Marshall, being of intermediate composition—$H_2O_2 : 2SO_3$. The members of such a series of persulphuric acids may be provisionally distinguished as "persulphuric," "perdisulphuric," and "pertetrasulphuric acids."

Evidence of the composition of the acid in question has been obtained by determining the equilibrium which results on mixing solutions of sulphuric acid and hydrogen peroxide in various proportions. Over fifty different mixtures have been examined, and it has been found that the variation of equilibrium as the concentration changes can be represented by an equation of the type—

$$\frac{c_1}{c_2} = k_1\left(\frac{c_3}{c_4}\right)^4 + k_2\left(\frac{c_3}{c_4}\right)^6$$

where c_1 and c_2 are the relative molecular concentrations of the "persulphuric" acid and hydrogen peroxide, whilst c_3 and c_4 are the relative molecular proportions of sulphuric acid and water in the mixture. The peroxidation is effected almost entirely within the limits H_2SO_4 and $H_2SO_4{,}5H_2O$. The results indicate that in addition to the "pertetrasulphuric" acid, a small but increasing proportion of "perdisulphuric" acid is formed as the concentration decreases, and qualitative tests made in accordance with Caro's directions leave no doubt that this is the case.

80. "*On Diphenyl- and Dialphyl-ethylenediamines, their Nitro-derivatives, Nitrates, and Mercurichlorides.*" By W. S. Mills, B.A.

When diphenylethylenediamine dissolved in glacial acetic acid is nitrated, a yellow precipitate is obtained towards the completion of the nitration. Purified from nitrobenzene, it melts at 307—308°, and is the *tetranitro-*

diphenylethylenediamine, $C_2H_4(NHC_6H_3(NO_2)_2)_2$, of Jedlicka (*Journ. Prakt. Chem.*, 1893, [ii.], xlviii., 202), prepared by the interaction of 2 : 4 bromodinitrobenzene and ethylenediamine.

Water added to the solution from which the last-mentioned compound has separated precipitates a yellow amorphous powder, very soluble in acetone, ethyl acetate, and acetic acid. It melts at 85°, and is an *isomeric tetranitrodiphenylethylenediamine*, which, when boiled with acetone, is converted into Jedlicka's isomeride.

Diorthotolylethylenediamine nitrated in a similar manner gives a solution from which water precipitates a yellow amorphous powder, which melts at 78—79°; it is a *tetranitrodiorthotolylethylenediamine*,—

$$C_2H_4(NHC_6H_2{\cdot}2NO_2{\cdot}CH_3)_2.$$

When heated with ethyl acetate, this compound undergoes a change similar to that observed in the case of its phenyl homologue, a yellow powder being obtained which melts at 178—180°, and is probably an *isomeric tetranitrodiorthotolylethylenediamine*. Dimetatolylethylenediamine on nitration gives a yellow powder very soluble in acetic acid, melting at 57°, and is *tetranitrometatolylethylenediamine*. Partial nitration of diparatolylethylenediamine gives a mass of fine orange-red crystals. This is *dinitrodiparatolylethylenediamine*, $C_2H_4(NHC_6H_3{\cdot}NO_2{\cdot}CH_3)_2$, melting at 195—196° and is identical with that prepared by Gattermann and Hager (*Ber.*, 1884, xvii., 778) by the interaction of ethylene dibromide and metanitroparatoluidine.

Diphenylethylenediamine nitrate,—

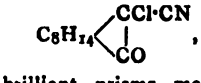
$$C_2H_4(NHC_6H_5)_2{\cdot}2HNO_3,$$

consists of needle-shaped crystals, m. p. 164·5°. *Diorthotolylethylenediamine nitrate*, $C_2H_4(NHC_6H_4CH_3)_2{\cdot}2HNO_3$, needles, m. p. 152°. *Diparatolylethylenediamine nitrate*, needles, m. p. 166·5° *Dimetatolylethylenediamine nitrate*, needles, m. p. 153°.

The mercurichlorides are prepared by adding alcoholic solutions of mercuric chloride to alcoholic solutions of the bases in molecular proportions, when crystalline precipitates separate slowly on standing, except in the case of the metatolyl compound, which is obtained by spontaneous evaporation of the mixed solutions. *Diphenylethylenediaminemercurichloride*, $C_2H_4(NHC_6H_5)_2HgCl_2$, well-formed rhombic plates, m. p. 129°. *Diorthotolylethylenediaminemercurichloride*, small rhombic plates, m. p. 110°. *Diparatolylethylenediamine mercurichloride*, in white feathery flakes, m. p. 133°. *Dimetatolylethylenediamine mercurichloride*, rosettes needles, m. p. 79—80°.

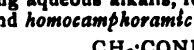
81. "*Derivatives of Cyanocamphor and Homocamphoric Acid.*" By ARTHUR LAPWORTH.

Cyanocamphor is easily obtained in large quantities by a modification of the method of Bishop, Claisen, and Sinclair (*Annalen*, 1894, cclxxxi., 351). *Chlorocyanocamphor*,—



crystallises in brilliant prisms melting at 98—100°. Cyanocamphor and its bromo- and chloro-derivatives are dimorphous.

When the halogen derivatives of cyanocamphor are heated with strong aqueous alkalis, reduction occurs, and cyanocamphor and *homocamphoramic acid*—

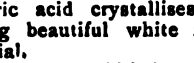


are produced. The latter crystallises in large prisms which melt and decompose at 209—210°.

Homocamphoric acid crystallises well from nitrobenzene, forming beautiful white needles which are apparently uniaxial.

When homocamphoric acid is heated with bromine in a sealed tube, action occurs at 140°, but much more than 1 molecular proportion of bromine is used up, and the products are difficult to isolate. When it is brominated in presence of phosphorus pentabromide and the product is treated with alcohol, diethylaniline, and alcoholic potash successively, a *lactone*, $C_{10}H_{16}O_2$, is obtained; this is volatile with steam, and crystallises in colourless needles melting at 146°.

α-Bromohomocamphoric acid,—



is made by heating the dichloride of homocamphoric acid with bromine on the water-bath. It separates from ethyl acetate in small, six-sided leaflets, and from benzene in slender needles. It melts sharply, under certain conditions, at 181—182°. Unlike all the allied substances, namely, camphoric acid and its halogen derivatives, and homocamphoric acid itself, it dissolves in chloroform and benzene.

When its diethyl ester is heated with diethyl aniline, it loses ethyl bromide, and gives the ethyl ester of *homocamphanic acid*,—

The same acid is obtained by warming an aqueous solution of sodium bromohomocamphoric acid; it crystallises in glistening needles or six-sided plates melting at 161—162°.

Experiments are being made with the object of preparing *dehydrohomocamphoric acid*,—

which should afford interesting results on oxidation.

(To be continued).

NOTICES OF BOOKS.

The Kinetic Theory of Gases. By Dr. OSKAR EMIL MEYER. Translated from the Second Edition, by ROBERT E. BAYNES, M.A. Pp. 472. London, New York, and Bombay: Longmans, Green, and Co.

DR. MEYER has made many additions to this work since its first appearance more than twenty years ago, though the general plan of the book remains the same.

The subject is treated in three parts, with six mathematical appendices; this arrangement facilitating the task of the ordinary reader who wishes to see how the several objections to the theory have been met, or attempted to be met; whilst for the chosen few who revel in mathematics, ample material has been provided at the end of the volume for the satisfaction of their appetites.

Part I treats of molecular motion and its energy, and in five chapters Dr. Meyer sets forth the history of the theory since its practical inception by Bernouilli in 1752, and its modern development by Clausius, Joule, and Maxwell, almost a century later. During the last half century, however, the range of conditions under which gases may be examined has been enormously increased, and from time to time objections have been raised against the theory as originally propounded, which have almost caused it to be finally rejected as untenable; but at the critical moment some one (chiefly German) has always arisen and proposed some slight modification of the original simple equations, taking account of some hitherto ignored factor, and once more reconciling theory with experimental results, until at the present day it rests upon hypotheses regarding the nature of ultimate matter, which, however they may appeal to the scientific imagination, seem for the present to be neither capable of being proved nor disproved.

As an illustration of the thorough manner in which

Dr. Meyer has explored his ground, it may be noted that in the chapter on "Maxwell's Law," which covers fifty pages of the text, there are no less than ninety-eight references to original publications bearing on the subject.

In Part 2, dealing with "Molecular Free Paths, and the Phenomena conditioned by them," fully as much progress has been made in the direction of a more searching enquiry into all the governing factors, with the result that many phenomena which, a quarter of a century ago, appeared to be at variance with theoretical requirements, have been recognised as agreeing with theory well within the limits of experimental error. The theory of the viscosity of gases is set forth with great clearness in Chapter 7, where the influence of partial or complete dissociation of a compound molecule on the viscosity is shown to fully account for the varying results obtained by different observers under different conditions.

In considering the theory of the conduction of heat, it should be noted that ethylene and nitrous oxide do not give nearly such good results as other gases, for in the tables on page 294 these gases show a difference of 10 per cent between the calculated and observed values, whilst air and carbonic acid show a difference of less than 1 per cent; it is only fair, however, to point out, as Dr. Meyer himself does, that the factors of the calculated figures are themselves approximations, and that "all differences between theory and observation might find their explanation in the accumulation of the errors of experiment."

Part 3, which deals with "The Direct Properties of Molecules," is much more speculative in character. In fact, as Dr. Meyer is careful to point out, many of the results arrived at may be accepted as not impossible rather than as probable.

When we consider, for instance, the several means by which the probable diameter of the "molecular sphere of influence" is calculated, each of which involves quantities that are only approximately known, it is a matter for surprise that Dorn, Loschmidt, van der Waals, and Meyer should have arrived at figures which lie within such close limits as 1/5th and 2 millionths of a millimetre, more especially since the larger figure is "a limit greater than which a molecule can not be."

The Mathematical Appendices, six in number, are of interest to those who can understand them, but do not appear to contain anything not previously published.

Physical Reactions and the "Mass Law." By Azariah T. Lincoln. (Reprinted from the *Journal of Physical Chemistry*, vol. iv., No. 3, March, 1900).

An interesting and useful contribution to our knowledge of a class of phenomena to which but little attention has been paid. Professor Bancroft in 1894 showed that the "Mass Law" could be applied to a class of conditions of equilibrium in which the exponential factors of the mathematical formula representing the equilibrium are not necessarily integers, and are usually not integers. This formula is empirical, and Mr. Lincoln has endeavoured to show by experiment that it is the mathematical expression of a new Law of Nature.

The ground to be covered is very wide and in general very complex. Mr. Lincoln has confined his experiments to one of the simplest class of phenomena, and, so far as he has gone, has succeeded in demonstrating the all but identity of the observed and calculated results.

The application of the mass law to the case of two non-miscible liquids and a consolute liquid is as follows:—If the amount of the consolute liquid be called "z" and the amounts of the non-miscible liquids "x" and "y," then—

$$\frac{x^{\alpha}y^{\beta}}{z^{\alpha+\beta}} = C.$$

Or, $\alpha \log x + \beta \log y - (\alpha+\beta) \log z = \log C = C'.$

Mr. Lincoln has performed a series of most careful experiments with benzol, water, and alcohol in various proportions, and has found that the calculated and observed quantities of alcohol, "the unknown quantity," agree within one-third of one per cent, thus showing in a remarkable way both his own skill as an experimenter and the accuracy of the empirical formula as applied to the phenomena under investigation.

The Theory of Electrolytic Dissociation and some of its Applications. By Harry C. Jones. London: Macmillan and Co., Lim. New York: The Macmillan Co. 1900. Pp. 289.

Many students have found a difficulty in obtaining a comprehensive account of the newer developments in physical chemistry, such as the relation between osmotic pressure and gas pressure, the origin and significance of the theory of electrolytic dissociation, &c. Until the appearance of this volume much of this information could only be obtained from certain original papers, not always easy of access: the above-mentioned and other matters are now placed before the reader in a systematic manner.

The author divides his subject into four parts or chapters. Chap. I., on the earlier Physical Chemistry, comprises a study of the relations between properties and composition, and properties and constitution; the development of thermo-chemistry and electro-chemistry, chemical dynamics and chemical statics, from which can be seen not only the state of development, but also the inherent nature of physical chemistry. The aim of the physical chemist in early times was to connect, and thus systematise, the great masses of isolated facts, and refer them as far as possible to common causes.

Pfeffer's osmotic investigations next claim attention in Chapter II. The phenomenon of osmosis is the passage of a pure solvent through a partition, to join a solution of a substance on the other side, which substance is unable to pass through the partition itself; the pressure thus produced is called osmotic pressure.

Chapter III. deals with the evidence bearing upon the theory of electrolytic dissociation; the evidence in favour of this theory is so overwhelming, in comparison with the few apparent exceptions, that the author considers these latter should be examined very closely before concluding finally that they are real exceptions.

The fourth and last chapter is on some applications of the theory of electrolytic dissociation, or, in other words, showing what is the scientific use of this theory. A careful study of this chapter will bring out a fact of profound significance. The theory, it will be seen, co-ordinates and correlates heterogeneous masses of facts, which apparently bore little or no relation to each other, and refers them to a common cause. Physical chemistry, the author tells us in conclusion, is furnishing us by the aid of the theory of electrolytic dissociation, with rational explanations of chemical processes whose meaning was entirely concealed, and is rapidly placing chemistry upon that exact mathematical basis which physics has so long enjoyed.

Kelly's Directory of Chemists and Druggists. 1900. Ninth Edition. London: Kelly's Directories, Lim. Pp. 691.

In the pages of this Directory will be found the names of over 49,000 persons, being an increase, as compared with the last edition, of about 9000. These include not only chemists and druggists, as the title might imply, but also manufacturing chemists, dentists, veterinary surgeons, wholesale druggists, drysalters, mineral water manufacturers, photographers, &c. Another addition is, that the names for the Isle of Man and the Channel Islands are included for the first time.

CORRESPONDENCE.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

Sir,—"A Ghost of 200 Years" appears to be acquainted with buried discoveries of the past; it would be more to the purpose if he could tell us whether the fact I stated were generally known now. The only suggestion that I have come across and acted upon more than once (with unsatisfactory results) is that of using freshly broken crystals of quartz (Miller, "Elements of Chemistry," vol. ii., p. 202). The use, however, of broken fragments of common tobacco-pipe leaves nothing to be desired, and would certainly have been mentioned in preference if it had been known to him. Fresenius suggests no foreign body, and Crookes does not notice this under the above heading. Is it because its use is so very common? There are many little details of manipulation which we hit upon after years of practice, only because they are not recorded in our text-books.—I am, &c.

G. F. M. Fielding.

Staplehurst, June 18, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 21, May 21, 1900.

Formation of Nitric Acid during Combustions.—M. Berthelot.—The formation of small quantities of nitric acid—or, more precisely, oxides of nitrogen transformable into this acid—during the combustion of hydrogen or various hydrocarbons, has been observed by many chemists, but has never been the subject of a systematic study. The author has undertaken this investigation, utilising his numerous determinations of heats of combustion and formation of organic compounds. During these determinations, the small quantity of nitric acid was always determined by reason of the simultaneous oxidation of the nitrogen contained in the oxygen used.

Limits of Combustibility of Hydrogen and Gaseous Hydrocarbons Diluted with Large Volumes of Air by means of Copper Oxide at a Red Heat.—Armand Gautier.—Hydrogen and gaseous hydrocarbons, when passed over copper oxide at a red heat, are transformed into water and carbon dioxide, but this reaction is retarded to a certain extent by the products themselves as they are formed; and, in the case of hydrogen or the other combustible vapours being mixed beforehand with large volumes of inert gas, the question arises as to whether their combustion is total. The author's first experiments were tried with hydrogen mixed with five thousand times its volume of air, and passing over a column of red-hot copper oxide 70 c.m. long at the rate of 2 to 3 litres per hour, and he finds that the hydrogen is entirely burnt. In the case of methane, he finds that the quantity of methane which burns when passing over copper oxide, together with large quantities of air, diminishes with the dilution.

Action of Hydrobromic Acid on Dextro-benzylidene Camphor, Monobromo-benzyl Camphor, Dextro-benzylidene-campholic and Phenyloxyhomo-campholic Acids.—A. Haller and J. Minguin.—When finely powdered dextro-benzylidene camphor is dissolved in crystalline acetic acid saturated with hydrobromic acid, and this is left standing in the cold for a month or six weeks, white crystals separate out, which have the formula $C_8H_{14}<^{CH.CH.Br.C_6H_5}_{CO}$, and are called monobromo-benzyl camphor. Various properties and reactions of this substance are described in this paper.

Anhydrous Calcium Dioxide and the Constitution of its Hydrates.—M. de Forcrand.—The precipitate of hydrated calcium dioxide, CaO_2+8H_2O, has been already described by the author. This substance, when kept cool, can be dried in a vacuum by means of phosphoric anhydride. After six days, a dry amorphous powder was produced, which gave on analysis $CaO_{1\cdot94}+0\cdot13H_2O$, or very nearly anhydrous CaO_2. The author examines the various hydrates which this substance forms, and measures the heat of formation.

Some Properties of Aluminium, and the Preparation of Gaseous Hydrogen Phosphide.—Camille Matignon.—The author uses aluminium phosphide for the preparation of hydrogen phosphide. The aluminium phosphide is placed with water in a flask, the cork being fitted with a dropping funnel containing sulphuric acid. Water attacks the phosphide at ordinary temperatures, but traces of acid accelerate this decomposition. By means of the dropping funnel the action can be regulated. The gas produced by this means is extremely pure. The actions of sulphur, selenium, phosphorus, arsenic, and antimony on aluminium powder are of explosive violence, and are strongly exothermic. This method of direct preparation is, however, very rapid and easy.

Compounds of Lithium Bromide with Ammonia Gas.—J. Bonnefoi.—In the same manner as lithium chloride, lithium bromide, when absolutely pure and dry, absorbs ammonia gas, and forms with it four compounds which are extremely deliquescent, $LiBr.NH_3$, $LiBr.2NH_3$, $LiBr_3NH_3$, and $LiBr_4NH_3$. The four molecules of ammonia successively added to LiBr evolve:—First molecule, +13·293; second molecule, +12·644; third molecule, +11·526; fourth molecule, +10·635; making a total of +48·098 calories, numbers in each case greater than the corresponding numbers for LiCl.

Two Polysulphides of Lead and Copper.—F. Bodroux.—When polysulphide of calcium is poured into a dilute solution of a lead or copper salt, a voluminous precipitate is produced, whose colour varies with the nature of the metal. The author isolates these two bodies, and shows by analysis that they have the composition PbS_5 and Cu_2S_5 respectively. PbS_5 is a purplish red substance at a low temperature. Above 10° it rapidly decomposes into lead monosulphide and sulphur. Cu_2S_5 is a brownish red body, stable at ordinary temperatures, but decomposing after some days into the monosulphide and sulphur.

A Chlorosulphide of Mercury.—F. Bodroux.—When calcium polysulphide acts on excess of a solid saturated solution of mercuric chloride, a totally different action takes place from those described in the previous papers A yellow precipitate is formed, whose colour gradually fades. When dried, it is a white powder. Analysis show. this substance to have the formula $Hg_2S_5.HgCl_2$. This chlorosulphide is very stable at ordinary temperatures, but superficially alters on prolonged exposure to light. It is insoluble in the alkaline sulphides and the alkalis, which blacken whilst decomposing it.

Action of Water on Mercurous Sulphate.—M. Gouy.—The author investigates the decomposition of mercurous sulphate by means of water, and the formation of basic mercurous sulphate.

Partial Synthesis of Lævo-erythrite.—L. Maquenne.—This preliminary notice contains an account of various new compounds which the author obtained in the course of the synthesis of lævo-erythrite: xylosoxime, acetyl-xylonic nitrile, *l*-erythrose-acetamide, and, finally, *l*-erythrite.

Preparation of the Dialcoylamidodichlor-anthraquinones.—E. C. Severin.—It appears that the presence

of two atoms of chlorine with regard to one of the carboxyl groups prevents the condensation of the dichlorodialcoylamidobenzoylbenzoic acid, and is only effectual when operating on the benzyl acids, and under other conditions than those described by MM. Haller and Guyot.

Monoiodohydrine of Glycol.—E. Charon and M. Paix-Séailles.

γ-Chlorocrotonic Acid.—R. Lespieau.

MISCELLANEOUS.

Society of Arts Medals.—The Council have awarded the Society's Silver Medal to the following readers of papers during the Session 1899-1900:—

To D. E. Hutchins, Conservator of Forests, Cape Town, for his paper on "National Forestry."

To Sir W. Martin Conway, M.A., for his paper on "Some of the Undeveloped Resources of Bolivia."

To Edmund Wilson, for his paper on "The Housing of the Poor."

To Professor R. W. Wood, for his paper on "The Diffraction Process of Colour Photography."

To Edwin Bale, R.I., for his paper on "Artistic Copyright."

To Miss Halsey, for her paper on "Some Unfamiliar Masterpieces of the Italian School."

To Professor W. M. Flinders Petrie, D.C.L., for his paper on "A National Repository of Science and Art."

To A. R. Colquhoun, for his paper on "Russia, Persia, and Afghanistan."

To Sir William Lee-Warner, K.C.S.I., M.A., for his paper on "Our Work in India in the 19th Century."

To Christopher Rawson, F.I.C., for his paper on "The Cultivation, Manufacture, and Use of Indigo—Position of the Industry in India."

To John Ferguson, for his paper on "Old and New Colombo."

To the Right Hon. Sir Charles Wentworth Dilke, Bart., M.P., for his paper on "The Century in Our Colonies."

To Cyril Davenport, for his paper on "Niello Work."

To Lasenby Liberty, for his paper on "English Furniture."

The Society of Arts Albert Medal for the present year has, with the approval of His Royal Highness the Prince of Wales, President of the Society, been awarded to Mr. Henry Wilde, F.R.S., "for the discovery and practical demonstration of the indefinite increase of the magnetic and electric forces from quantities indefinitely small." This principle is the one on which the invention of the modern dynamo machine is based, and is employed in all modern dynamos.

Action of Hypochlorous and Hypobromous Acids on Acetylene, and on the Mono-substituted Acetylenes.—N. Vittorf.—Acetylene does not unite with HClO up to 50°, but it unites with 2HBrO, forming dibromoacetic aldehyde, $CHBr_2.CHO$: this latter gives, besides the known monohydrate, a stable crystallised dihydrate. These hydrates, the same as the aldehyd, give glyoxime by means of the chlorhydrate of hydroxylamine. The union of allylene with HClO or with HBrO produces a dichlorised or dibromised ketone, which is transformed into methylglyoxime by the chlorhydrate of hydroxylamine. By the combination of HClO or HBrO with trimethylallylene, we obtain the dichlorised or dibromised pinacolines, the first fusible at 51° and the second at 75°—75·5°. With phenyl-acetylene HClO gives dichloroacetophenone, fusible at 19°—20°, which furnishes a dioxime fusible at 156°—158°.—*Journ. Soc. Phys. Chim. R.*, xxxi., p. 490.

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MATRICULATION EXAMINATION.

A Matriculation Examination will be held at the University during the week commencing Monday, September 17th, 1900. The last day for receiving applications for entry will be September 1st.—For further particulars of the subjects of examination and fees, &c., apply to the Registrar of the University.

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W. E. R. ALLEN,
Clerk of the Joint Committee.

Glamorgan County Offices,
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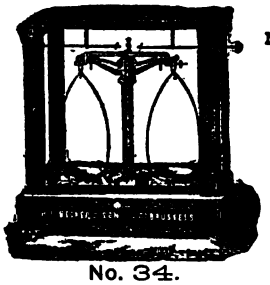

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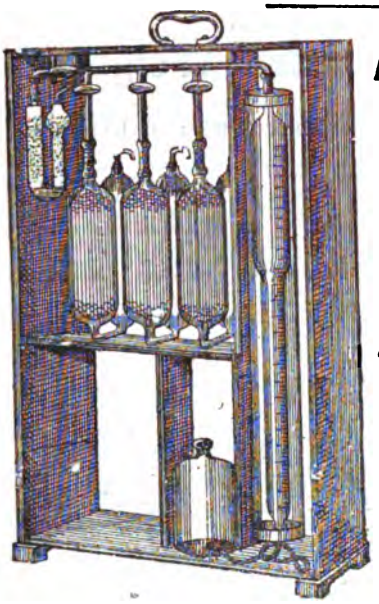

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Edited by Sir Wm. Crookes, F.R.S.] (WITH WHICH IS INCORPORATED THE "CHEMICAL GAZETTE") [Established Fifty-three Years.

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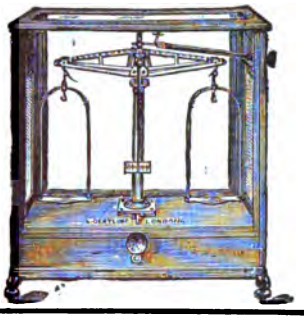

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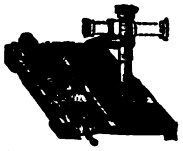

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THE CHEMICAL NEWS.

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THE NATURE AND ORIGIN OF THE POISON OF *LOTUS ARABICUS*.*

By WYNDHAM R. DUNSTAN, M.A., F.R.S., Sec.C.S., Director of the Scientific Department of the Imperial Institute, and T. A. HENRY, B.Sc., Lond.

Lotus Arabicus is a small leguminous plant resembling a vetch, indigenous to Egypt and Northern Africa. It grows abundantly in Nubia, and is especially noticeable in the bed of the Nile from Luxor to Wady Halfa. It is known to the natives as "Khuther," and old plants with ripe seed are used as fodder. At certain stages of its growth it is highly poisonous to horses, sheep, and goats, the poisonous property being most marked in the young plant up to the period of seeding. Owing to the trouble which this plant has given to the military and civil authorities in Egypt, the assistance of the Director of Kew was sought in order that the precise nature of the poison might be ascertained, and, if possible, a remedy found. The matter having been referred to the Scientific Department of the Imperial Institute, Mr. E. A. Floyer, Director of Egyptian Telegraphs, collected some of the material for investigation. The dried plant is unusually green, and possesses the aroma of new-mown hay.

It was found that when moistened with water and crushed, the leaves of the plant evolved prussic acid in considerable quantity, the amount being greatest in the plant just before and least just after the flowering period. Further investigation has shown that the prussic acid originates with a yellow crystalline glucoside ($C_{22}H_{19}NO_{10}$), which it is proposed to name *lotusin*. Under the influence of an enzyme, also contained in the plant, lotusin is rapidly hydrolysed, forming *prussic acid*, *sugar*, and a new yellow colouring matter (*lotoflavin*). The hydrolysis may be effected by dilute acids, but it is only very slowly brought about by emulsin and not at all by diastase. The peculiar enzyme, which it is proposed to call *lotase*, appears to be distinct from the enzymes already known. Its activity is rapidly abolished by contact with alcohol, and it has only a feeble action on amygdalin. Old plants are found to contain lotase but no lotusin.

The *sugar* has been proved to be identical with ordinary dextrose.

Lotoflavin, the yellow colouring matter, has the composition expressed by the formula $C_{15}H_{10}O_6$. It belongs to the class of phenylated pheno-γ-pyrones, and is a dihydroxychrysin, isomeric with luteolin, the yellow colouring matter of *Reseda luteola*, and with fiseti in the yellow colouring matter of *Rhus cotinus*.

The decomposition which ensues on bringing lotase in contact with lotusin, as happens when the plant is crushed with water, is therefore probably expressed by the following equation:—

$$\underset{\text{Lotusin.}}{C_{22}H_{19}NO_{10}} + 2H_2O = \underset{\text{Lotoflavin.}}{C_{15}H_{10}O_6} + \underset{\text{Prussic acid.}}{HCN} + \underset{\text{Dextrose.}}{C_6H_{12}O_6}.$$

Hydrocyanic (prussic) acid occurs in small quantity in many plants, and according to Treub and Greshof is often present in the free state. The only glucoside at present definitely known which furnishes this acid is the well-known amygdalin of bitter almonds, which under the influence of the enzyme emulsin, also contained in the almond, breaks up into dextrose, benzaldehyde, and prussic acid.

Owing to the scientific interest which attaches to this new glucoside, its properties and those of its decomposition products have been very fully studied, and the characteristics of the new enzyme have also been investigated.

We are much indebted to Mr. Floyer for the great pains he has taken to collect, in Nubia, the necessary material for this investigation, and also to Sir W. T. Thiselton-Dyer for having grown the plant at Kew, from seed obtained from Egypt.

* Abstract of a Paper read before the Royal Society, June 14, 1900.

ON THE EXISTENCE OF THERMAL CENTRES OF STABILITY IN COMPOUNDS.

By GEOFFREY MARTIN.

All chemical combinations may be divided into two great classes—endothermic and exothermic.

In endothermic compounds the atoms are held together in such a way that energy is stored up in the molecule. Consequently, when the molecule is decomposed, this stored-up energy is liberated, principally in the form of heat. Endothermic compounds are usually explosive, and for the reason that, in separating the atoms of the molecule, work is done *with* not *against* the internal atomic forces.

In exothermic compounds, however, the contrary is the case, and the coming together of the atoms which compose them is attended with the evolution of energy. In order to decompose such a molecule, work must be done *against* the atomic forces within the molecule. Such compounds are therefore usually stable.

The heat of formation of a compound may be either positive or negative, according as it is exothermic or endothermic. Now, it is well known that the heat of formation of a compound is not a constant, but varies with the temperature. In a great many cases it diminishes with the increasing temperature. This may also be directly deduced from the kinetic theory of matter (in the case of exothermic compounds at least), for the higher the temperature the greater the internal atomic motion within the molecule, consequently the more feebly are the atoms held together. But the force with which the atoms are held together must be some function of the absolute heat of formation of that compound; and, consequently, we should expect that, in the case of an exothermic compound, an increase in temperature should lead to a diminution in its heat of formation.

This reasoning assumes that the fundamental attraction of the atoms for each other is a constant—an assumption that is by no means warranted by the facts, as will be seen later.

The dependence of the heat of formation upon the temperature has been investigated in the case of very few endothermic compounds. Victor Meyer, however, in 1893, studied the case of hydrogen iodide. The heat of formation of this compound is −6100 calories. With increase of temperature, the negative value of its heat of formation continually diminishes until, at 324° C., it becomes zero. At still higher temperatures it becomes positive. In this case, then, we have an example of an endothermic compound gradually changing into an exothermic compound. Suppose, now, we heated an exothermic compound beyond the temperature at which its heat of formation is zero, would it conversely become an endothermic compound? I shall bring forward some indirect evidence later tending to show that, if this be so, we have the key wherewith to explain some very remarkable phenomena.

I fail to perceive the necessity of drawing an arbitrary distinction between endothermic and exothermic compounds. I would regard them as only the different stages of the physical alteration of a compound with the temperature. The fact that a compound is at ordinary temperatures endothermic or exothermic, as the case may be,

is, according to my way of looking at the matter, a mere accident of the temperature. A compound probably passes successively with the temperature from an endothermic to an exothermic state, and—at still higher temperatures—back again into the endothermic state. According to this view, the positive or negative value of heat of formation of a compound is a periodic or recurring function of the temperature. Of course, the body need not necessarily be regarded as capable of existing undissociated throughout all these changes of temperature.

According to the modern theory of chemistry, the dissociation of a compound is a reversible chemical reaction. If, therefore, we assume that the positive or negative value of the heat of formation of certain compounds is a periodic or recurring function of the temperature, we can apply the laws of thermodynamics to investigate in what manner the dissociation will vary with the temperature.

Consider the gaseous reaction—

$$A + B = A' + B';$$

where A and B are molecular species which, by reacting on each other, give rise to the molecular species A' and B'. Then, according to the law of mass action, the velocity of the change from the left to the right, in the sense of the above equation, is—

$$v = k \cdot c_1 c_2 \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1).$$

where c_1 and c_2 are the active masses of the reacting molecules A and B respectively, and k is the proportional constant. Similarly, the velocity of the reaction in the sense of the equation from the right to the left is—

$$v_1 = k' \cdot c_1' c_2' \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (2).$$

But when a state of equilibrium has been arrived at—

$$v = v_1$$

or—

$$k c_1 c_2 = k' c_1' c_2',$$

denote—

$$\frac{k}{k'} \text{ by K.}$$

Then—

$$K = \frac{c_1' c_2'}{c_1 c_2} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3).$$

K is the coefficient of equilibrium of the system, and it denotes how much faster the reaction proceeds in the sense of the above equation from the left to the right than from the right to the left.

This coefficient K varies with the temperature. This variation at constant volume is expressed by the equation derived from thermodynamical considerations by van 't Hoff—

$$\frac{d \log K}{d T} = \frac{q}{R T^2} \quad . \quad . \quad . \quad . \quad . \quad (4).$$

T denotes the absolute temperature of the system, while R is the ordinary gas constant in the equation $pv = RT$ expressed in suitable units. It is advisable to thoroughly understand the nature of q. q denotes the total internal change in the energy of the system in consequence of the reaction.

If the molecular species A and B when reacting to produce A' and B' *evolve* heat, then the products of the change (A' and B') possess *less internal energy* than the molecular species whence they are derived, namely, A and B. q is in this case associated with a *diminution* in the total internal energy of the whole system, and consequently is *negative*. If, however, the products A' and B' are produced from A and B with the absorption of heat, then the products A' and B' are at a higher energy potential than A and B; consequently, the reaction A + B = A' + B' is associated with an *increase* in the total internal energy of the system, and it follows that q is *positive*. To sum up then, q is negative in the case of an exothermic reaction, and positive in the case of an endothermic reaction.

Re-arranging Equation 4 we obtain—

$$d \log K = \frac{q}{R} \cdot \frac{d T}{T^2},$$

integrating—

$$\log K = -\frac{q}{R} \cdot \frac{1}{T} + \text{constant}.$$

Let K_1 and K_2 represent the equilibrium coefficients of the system at two different temperatures T_1 and T_2 ($T_2 > T_1$). Further, suppose T_2 and T_1 to be close together, so that q will not change noticeably between the interval T_2 and T_1. Then, substituting these values in the above equation and subtracting, we obtain—

$$\log K_2 - \log K_1 = \frac{q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right). \quad . \quad (5).$$

Now, according to our hypothesis, q successively changes from a positive value (say) through zero to a negative value.

So long as r has a finite *positive* value, the expression $\log K_2 - \log K_1$ has also a positive value. Hence—

$$K_2 > K_1$$

so long as the value of the equilibrium coefficient increases with the increasing temperature—although at a diminishing rate, as may be seen from the form of Equation 5. But K represents how much faster the system A + B reacts to produce A' + B' than the system A' + B' reacts to reproduce A + B.

Consequently an increase in the value of K means that there occurs in the gaseous mixture we are considering a *decrease* in the amount of A + B and an *increase* in the amount of A' + B' present per unit volume. Consequently, with rise of temperature, q remaining positive, the reaction—

$$A + B = A' + B'$$

tends more and more to cause the complete conversion of the A and B molecular species into the A' and B' species. Hence in those cases of dissociation which are attended by an *absorption of heat*—such as the dissociation of $CaCO_3$, N_2O_4, &c.—the dissociation becomes more and more complete as the temperature increases.

When, however, $q = 0$,

$$K_2 = K_1,$$

and so long as q retains this value a further increase of temperature does not cause the relative amounts of the species A and B, A' and B', in the system to vary.

But if q becomes negative, *i.e.*, if the reaction A + B = A' + B' is attended with the *evolution of heat*, as is the case when hydrogen and oxygen are heated together to a suitable temperature, then—

$$\log K_2 - \log K_1 = \text{a negative quantity}.$$

That is—

$$K_2 < K_1.$$

Hence with increasing temperature the system A + B reacts to produce A' + B' at a *slower rate* than the system A' + B' reacts to reproduce the system A + B.

Consequently, with increase of temperature the amount of the molecular species A and B in the system continually increases at the expense of the A' and B' molecular species.

We thus arrive at the following law, which is known as van 't Hoff's *Principle of Mobile Equilibrium*:—

If we heat a chemical system at constant volume, then there occurs a displacement of the state of equilibrium, and in that direction towards which the reaction advances with the absorption of heat.

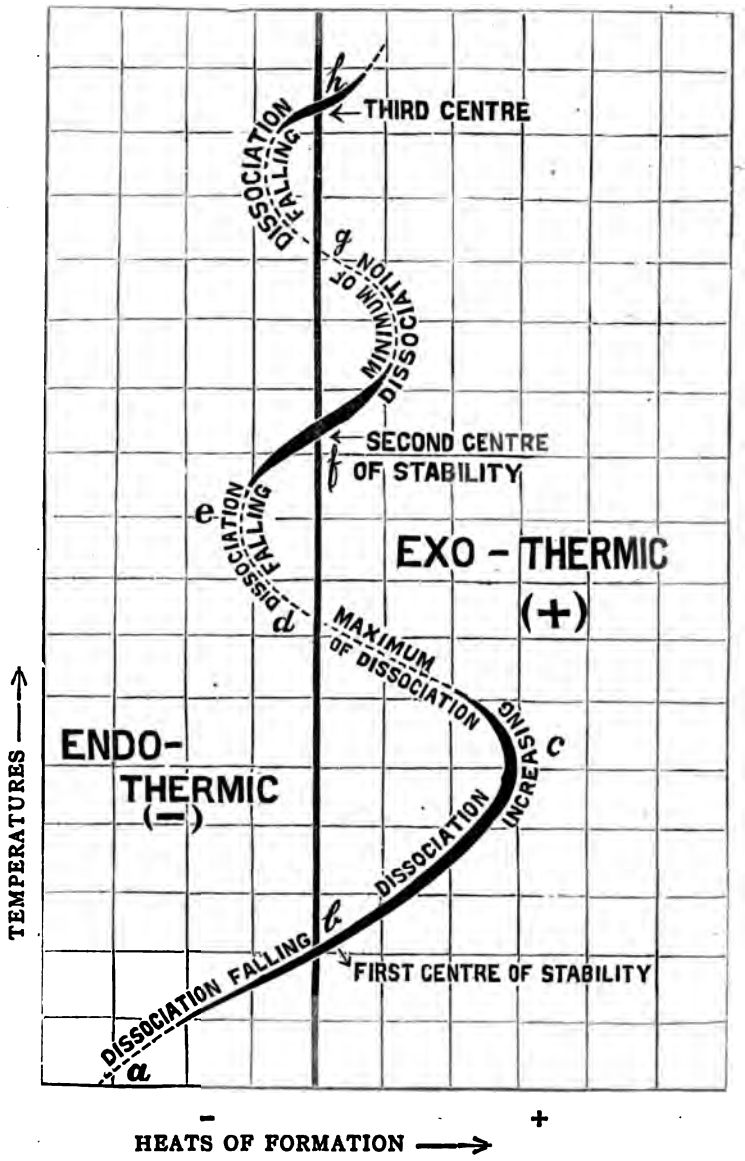
Assuming the truth of the supposition that the heat of formation of compounds is a periodic or recurring function of the temperature, we may draw such a hypothetical curve representing this variation in the value of q as is shown in Fig. 1.

The abscissæ represents the heats of formation of the compound, and the ordinates the temperature.

On applying van 't Hoff's "Law of Mobile Equilibrium," we deduce that from *a* to *b* the dissociation of the compound continually retreats. At the point *b*, where the heat of formation is zero, a point of minimum dissociation occurs. This point is therefore the first centre of stability of the compound.

On further increasing the temperature the dissociation will continually increase, and the compound becomes exothermic. But it will be noticed that there are now gas. On further heating, however, the heat of formation gradually diminishes, and consequently the increased motion of the atoms causes an increase in the amount of dissociation, which increases continually from (say) *c* to *d*, and attains a maximum at *d*. In many cases the body is completely decomposed at *d*; for example, silicon hexchloride.

At *d* the heat of formation again becomes zero, and further heating causes *q* to become negative. Conse-



two opposing forces at work. From *b* to *c* the compound becomes more and more exothermic, and consequently more and more stable. Nevertheless, with the increase of temperature, the motion of the atoms in the molecule becomes more pronounced. Usually the increased attraction of the atoms for each other is greater than their tendency to fly apart, and consequently from *b* to *c* the dissociation in many cases becomes practically negligible. Examples of such stable gases are ammonia and hydrochloric acid

quently there is now a tendency for the atoms to aggregate together again, and this tendency increases continually with the temperature until the point *f* is reached. At *f* the dissociation will again be a minimum, and there will result a second "centre of stability" of the compound. By following up the curve we obtain a third centre of stability at *h*.

Of course one can hardly expect a gaseous body to exist all along the curve. In most cases it would probably only

be capable of existing at certain points. The dotted part of the curve shows when the body would be most liable to be incapable of existing.

In all cases, however, we should look for the substance to appear at the points *b*, *f*, and *h*, which we will call the first, second, and third centres of stability of the compound. On searching for experimental evidence of the existence of these points, I have collected at least six instances. Under this heading, of course, I include recurrent chemical reactions.

1. *The case of Silicon Hexchloride*, Si_2Cl_6.—This substance is a colourless volatile liquid, which boils at 146° C. without decomposition. At 350° C. the vapour commences to dissociate, and the amount of dissociation continually increases up to 800° C., when the compound is completely resolved into silicon and the tetrachloride, thus:—

$$2Si_2Cl_6 = 3SiCl_4 + Si.$$

Troost and Hautefeuille found, however, that if the temperature be quickly raised above 1000° C. that no dissociation occurred. The substance, therefore, is stable above 1000° C. and below 350° C., but yet is incapable of existing at intermediate temperatures (*vide* Roscoe and Schorlemmer's "Chemistry").

2. *Hydrazine Hydrate*, $N_2H_4.H_2O$.—This substance is a strongly refractive fuming liquid, which possesses extremely active chemical properties. It boils, unaltered, at 118·5° C. under 739·5 m.m. pressure, and its vapour at 100° C. has, under diminished pressure, a density corresponding to the formula $N_2H_4.H_2O$. At 170° C. this vapour is completely dissociated into N_2H_4 and H_2O. At still higher temperatures the vapour density again continually increases, until finally it corresponds to a formula double that observed at 100° C. (*vide* Roscoe and Schorlemmer's "Chemistry").

3. *Ozone*, O_3.—This compound is at ordinary temperatures very unstable, and is completely decomposed into oxygen gas at 250° C. Nevertheless Dewar showed ("Roy. Instit. Year Book," 1887-89, p. 559) that it could be produced at a white heat. His method was to heat up a platinum cylinder to a white heat, and pass a stream of oxygen gas through the tube. The oxygen gas thus heated was suddenly withdrawn and cooled by being sucked down by a stream of cold water circulating in an interior platinum tube. The resultant gas is said to have contained a large amount of ozone. From this experiment Dewar concludes that ozone has two centres of stability, and one of these is near the melting-point of platinum.

4. *Hydrogen Selenide*, H_2Se.—This was investigated by Ditte in 1872 (*Comptes Rendus*). He showed that its dissociation first increased, then diminished until a minimum was reached, and then again increased.

5. According to Ostwald ("Outlines of General Chemistry," p. 312, 1890 ed.) cyanogen and acetylene are other examples. Both gases are formed in quantity at the highest temperature we can produce—in the electric arc and in the blast furnace. Yet at a red heat these gases cannot exist, cyanogen, as is well known, falling apart into its elements, whilst acetylene decomposes into other and more stable hydrocarbons, such as benzene.

6. Victor Meyer and Langer have shown that chlorine violently attacks platinum at a low temperature, but is without action upon it between the range 300°—1300° C. It then again begins to attack the platinum, the attack becoming violent between 1600°—1700° C. ("Brit. Assoc. Reports," 1885, p. 961).

These cases, be it noticed, were all discovered by accident. No systematic search has, so far as I am aware, been specially made to discover other examples of recurrent chemical changes.

An analogous phenomenon is presented by certain cases of electrolytic dissociation. Thus, phosphoric and hypophosphorous acids at the temperatures 54° and 75° C. respectively show *maxima of conductivity*. Above these temperatures they have negative temperature coefficients. It was Arrhenius who first discovered such electrolytes, and they are commonly regarded as anomalous. I would suggest, however, that they are but examples of a much wider law, and that probably under different physical conditions all compounds would exhibit analogous variations.

It must not be forgotten that hydrogen gas was once regarded anomalous in respect to its behaviour under pressure. But the subsequent researches of Amagat showed that under suitable physical conditions all the other permanent gases behaved similarly.

By glancing at the curve shown in Fig. 1, it will be seen that the smaller the maximum heat of formation of a compound is, the closer together will be the centres of stability.

In order, therefore, to experimentally investigate the recurring existence of compounds (or chemical reactions) at high temperatures, it would be well to choose bodies having small heats of formation.

Let not the significance of the above be missed. If there really exist thermal centres of stability, this proves that the attraction of the atoms for each other is not a constant, but depends upon their degree of relative motion; and, further, that this alteration in the attraction is a periodic function of the motion of the atoms.

I intend shortly to make an extensive research on the behaviour of bodies at very high temperatures.

Merchant Venturers' College, Bristol,
May 16th, 1900.

ON THE ALLEGED TRANSFORMATION OF PHOSPHORUS INTO ARSENIC.

By CLEMENS WINKLER.

In the March number of *Leopoldina*, No. 36, iii., page 40, F. Fittica seriously asserts that he has succeeded in partly transforming amorphous phosphorus into arsenic by heating it with ammonium nitrate to a temperature of, at most, 200°. He comes to the conclusion that arsenic is a nitrogen-oxygen compound of phosphorus, of the formula PN_2O, and that the "synthesis" takes place in accordance with the following formula:—

$$2P + 5NH_4.NO_3 = (PN_2O)_2O_3 + 10H_2O + 3N_2.$$

The assertion is not feasible. Arsenic has been obtained technically, especially in the form of its compounds, for at least 1000 years, and has been transformed on a very large scale from one form of combination into another in manufacturing operations without the least sign having hitherto been obtained which might justify any doubt of its elementary nature. There can be no question whatever that arsenic is really an element in the modern sense, a substance which cannot be decomposed into unequal constituents, and which cannot be composed from other elements. Fittica's assertion is based upon an enormous error, and it is to be regretted that this has been publicly expressed. But as it has been done, nothing remains but to correct it.

Fittica is not able to state exactly the amount of arsenic contained in the phosphorus which he used in his experiments, because he found it very variable—from 0· to 2·64 per cent—according to the use of different oxidants, or to that of yellow or red phosphorus. The highest proportion of arsenic was found in red phosphorus, by oxidising it with nitric acid, while no arsenic could be discovered if the solution had been effected by barium peroxide and sulphuric acid. Fittica only roughly estimates that phosphorus had been transformed to the extent of from 8 to 10 per cent into arsenic, and a foundation for the above equation expressing the transformation, and the extraordinary formula PN_2O for the "synthetically produced" arsenic is quite wanting.

It has long been known that commercial phosphorus is often contaminated with arsenic. The arsenic is derived

from the sulphuric acid used for the decomposition of the calcium phosphate, and its percentage has been stated by Wittstock (*Pogg. Ann.*, xxxi., 126) at 0·76. At present it may be higher, because sulphuric acid is no longer prepared, as it was then, from sulphur, but almost without exception from arsenical pyrites. Hjelt (*Dingl. Pol. Journ.*, ccxxvi., 174) found in a chamber acid prepared from Rio Tinto pyrites 0·202 per cent of arsenic. If it be assumed that acid of this nature were used for the preparation of phosphoric acid, and that the total contents of arsenic went into the phosphorus obtained, the latter would contain 1·87 per cent of arsenic.

The determination of the amount of arsenic offers no difficulties whatever, but it, of course, requires the carrying out of proper analytical methods. The precipitation of arsenic by sulphuretted hydrogen may easily fail if the arsenic is present as arsenic acid, or if the solution contains other oxidising substances, like nitric acid, hydrogen peroxide, &c. But, as is well known, it easily succeeds on properly treating an acid solution of arsenious acid with sulphuretted hydrogen. On repeating the experiment described by Fittica, oxidising 2 grms. amorphous phosphorus with 12·9 grms. of ammonium nitrate, by carefully fusing them together, according to his prescription, it was found that it is not easy to properly moderate the reaction, but that, nevertheless, or perhaps more correctly in consequence of it, a small amount of phosphorus escapes the oxidation. The melt contains, in consequence, the arsenic present in the phosphorus, to a great extent in the form of arsenious acid, and its aqueous solution yields, therefore, with sulphuretted hydrogen, an immediate yellow precipitate of arsenic trisulphide. This is also the case if one operates in a manner which appears to me preferable, namely, by not adding the total quantity of amorphous phosphorus to the ammonium nitrate, but to fuse the latter in a large porcelain crucible at 170° C., and then to add the amorphous phosphorus in small portions, rapidly mixing them with a thermometer. Very soon evolution of gas takes place, and the temperature rises, but the reaction is quiet and free from danger, provided the source of heat is immediately removed when a temperature of from 180° to 190° has been reached. As soon as the phosphorus has disappeared, the temperature is allowed to fall, a second portion is added, and so on, until the operation is finished, which for 2 grms. of phosphorus requires about three hours. In proportion to the formation of phosphoric acid the melting-point of the mass falls, so that at the later stages the addition of phosphorus may be made at 140°, but the temperature is always again raised during the stirring to 170°, when it spontaneously rises to 180—190°. In this way the operation is almost quantitative, although small losses by spurting cannot quite be avoided. The reaction appears mainly to take place according to the equation,—

$$2P+5NH_4NO_3=2H_3PO_4+7H_2O+10N,$$

but as soon as the phosphoric acid begins to be in excess the reaction is influenced by it, a feeble odour of nitric acid being observable. The fumes which are formed in small amount are probably nothing else but volatilising ammonium nitrate.

The melt thus obtained was easily soluble in water, and the solution had a strongly acid reaction. It was made ammoniacal, and the arsenious acid oxidised into arsenic acid by hydrogen peroxide.

For comparison, three portions of 2 grms. each of the same sample of phosphorus, which had been previously washed with water and dried at 110°, were oxidised:—

(*a*). By heating with a mixture of 30 c.c. nitric acid of 1·185 sp. gr. and 10 c.c. of water.

(*b*). By passing chlorine into water wherein the phosphorus was suspended till the fluid was saturated, and subsequent removal of the excess of the gas by continued gentle heating.

(*c*). By heating with a solution of 10 grms. of purest sodium hydroxide, and occasional addition of hydrogen peroxide, and subsequent acidulation of the solution with sulphuric acid.

The whole of these solutions were rendered ammoniacal, each evaporated to as small a volume as possible, and mixed with somewhat more than the calculated amount of an ammoniacal solution of ammonium magnesium chloride, of which 1 c.c. corresponded to 0·02 grm. of phosphorus. After the bulk of the precipitate had gone down more ammonia was added, and the mixture allowed to stand for forty hours. The washing of the precipitates was effected in the usual manner, with a mixture of one volume of ammonia to three volumes of water; no arsenic could be detected in the filtrate. The washed precipitates were dissolved in dilute sulphuric acid, and the solutions heated with repeated addition of sulphurous acid, and the excess of the latter expelled. They were then precipitated after dilution with sulphuretted hydrogen and allowed to stand overnight. The excess of sulphuretted hydrogen was mostly removed by gentle heating, but not entirely so; the arsenic trisulphide, which appeared to be of the same amount in all cases, was filtered off, dissolved on the filter in dilute warm ammonia, and the solution evaporated on the water-bath to dryness. The arsenic sulphide was then oxidised by nitric acid into arsenic acid, and this determined in the usual manner by precipitation with magnesia solution.

The amount of arsenic contained in the phosphorus was found to be as follows:—

	Per cent.
By oxidation with ammonium nitrate ..	1·910
,, ,, nitric acid	1·925
,, ,, chlorine	1·920
,, ,, hydrogen peroxide ..	1·920

The percentage of arsenic was therefore found to be the same in all cases; rather too low by oxidation of the phosphorus with ammonium nitrate, because a small loss could not be avoided. It follows that there can be no question that the conversion of phosphorus into arsenic in the manner described—and one safely can say in any manner whatever—is out of the question, and that Fittica's statement is based upon an error.

It must be admitted that this matter, which I have discussed most unwillingly, involves a very serious consideration. It would appear as if in inorganic chemistry a dangerous tendency showed itself of late to enter into speculations without those careful investigations which have hitherto distinguished German chemists. Cases are multiplying which tend to show that a theory is first formed, and that then one seeks to find what one wishes to find, or that, in the words of the physiologist Czermak, one starts from "erroneously observed facts," and thus falls into mistakes. The reason must be sought, to a great extent, in the fact that the art of analysis is being regrettably neglected. I intentionally say the "art," for between analysing and analysing there is the same difference as between artists' and stonemasons' work. One cannot expect analytical aptitude from the physicist, whose field of investigation begins more and more to extend to inorganic chemistry as electrolysis developes, and he, within his field of investigation, is able to discover useful, even great facts. But physical chemistry is in no way synonymous with inorganic chemistry; for the latter, far from being a finished department of science, embraces problems in infinite number which must be solved upon an entirely different road than that indicated by the ion-theory. The really successful carrying out of inorganic chemical investigations is only possible to him who is not only a theoretical chemist, but also an accomplished analyst; not only a practical, mechanical workman, but a thinking and forming artist, who clearly sees the theories of the operations, to whom the knowledge of proportion is a part of himself, and who in all his doings is led by a sense of order and neatness, but especially by a desire for truth.—*Berichte*, xxxiii., No. 10, 1693.

Freiberg, Saxony, May 26th, 1900.

AN IMPROVED DOOR FOR LABORATORY GAS FURNACE.

By B. A. BURRELL, F.I.C.

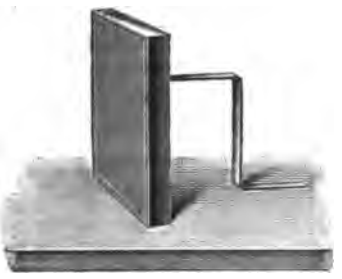
THE existing forms of doors for reverberatory gas furnaces are made of fire-clay or other refractory material, and have no outer casing; they are therefore easily broken, and not suited to withstand the rough usage they often receive from students.

Careless handling with the tongs soon causes the front projection to crumble away, and when this occurs they are awkward to move, and soon acquire a tendency to lean away from the furnace, leaving a gap between the furnace and the door, with consequent loss of heat. Further, all the existing forms of door, except Holloway's pattern, will not stand upright when removed from the furnace.

To overcome these objections I have devised the door shown in the accompanying cut:—

These doors have an outer casing of thin wrought-iron, except at the front, which is exposed to the interior of the furnace. At the front the outer casing only projects slightly round the four edges.

The bottom edge of the door is set at such an angle as to give the door a very slight backward tilt, but it is prevented from falling by the piece of bent hoop-iron inserted in a slot fixed on the back of the door; the door will thus stand upright when withdrawn from the furnace, and the hoop iron serves as a handle. (A piece of asbestos cloth should be used as a holder).



The two side edges of the casing are inclined at a slight angle from front to back, so that the front is slightly narrower than the back. Better fitting is thus obtained, and the lining of fireclay prevented from falling out.

The back of the iron casing is perforated with a number of small holes to permit of the more ready drying of the fire-clay mixture with which the door is lined. A suitable mixture for this purpose is composed of two-thirds raw fire-clay and one-third burnt fire-clay (by measure). A little sodium silicate added to the clay paste is advantageous.

The new door possesses the following advantages:—

1. It is practically indestructible, since the fireclay lining may be replaced at a merely nominal cost.
2. It can be adjusted so that it has no tendency to lean away from the furnace.
3. It stands upright when removed from the furnace.
4. It is cheaper than the furnace doors at present on the market.

An extended trial in this laboratory has resulted in the replacing of the plain fire-clay doors by the iron-bound doors, and the consequent abolition of a serious item of expense caused by frequent breakages.

The new doors may be obtained of Messrs. John J. Griffin and Sons, who supply them in sizes to fit their Holloway Gas Furnace or other sizes as required.

Metallurgical Laboratory,
Leeds Technical School.

SYNTHESIS OF SOME ORGANIC COMPOUNDS BY MEANS OF FERRIC CHLORIDE.

By N. MEISSEL.

THE condensation of *m*-xylene with chloride of acetyl in the presence of ferric chloride commences readily in the cold; it is finished by heating for twenty minutes on the water-bath; the product is then washed with water, and collected by the help of steam. In this manner we obtain dimethylacetophenone, $CH_3.CO.C_6H_3(CH_3)_2$, in the form of a liquid having an agreeable smell and a density of 1·0121 at 15°; its boiling-point is 227—228°. A small quantity of *o.p*-xylylic acids is also formed. The return is 30 per cent, but if we make use of a sulphocarbonic solution we can obtain as much as 75 per cent. In the same manner the author has prepared dimethylbenzophenone boiling at 362° (a return of 70 per cent), cymylmethylketone, $CH_3C_6H_3(COCH_3).CH(CH_3)_2$, a liquid of 0·9715 density at 15°, boiling at 240—242°, and giving a red colouration with ferric chloride in alcoholic solution. The tertiary chloride of butyl does not condense with *p*-xylene in the presence of ferric chloride; on the other hand, chloride of acetyl reacts on *p*-butyltoluene (tertiary) readily in the cold, giving rise principally to butylmethylacetophenone (72 per cent), an oily liquid with an agreeable odour, insoluble in water; $d = 0·9541$ at 15°; the alcoholic solution is coloured red by ferric chloride. The condensation of tetrachloride of carbon with benzene gives, uniquely, chlorised triphenylmethane, $Cl.C(C_6H_5)_3$, which is transformed by water into triphenylcarbinol; with chloroform and benzene we obtain a mixture of carbinol and triphenylmethane (we use 1 part of chloroform, 1 part of ferric chloride, and 6 parts of benzene, and heat on the water-bath). The condensation of the chlorocarbonate of iso-amyl with phenol gave *p*-isoamylphenol, fusible at 93—94°; in the same manner, with the chlorocarbonate of ethyl, we obtain *p*-ethylphenol.—*Berichte*, xxxii., p. 2419.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MAY 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, June 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from May 1st to May 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month of May shows that the drought still continues. The actual fall was 1·33

inches; the average fall is 1·83 inches; this leaves a deficit of 0·50 inch for the month, and reduces the previous excess for the year to 0·34 inch, or 3·8 per cent on the thirty years' average.

Our bacteriological examinations of 557 samples have given the results recorded in the following table; we have also examined 36 other samples, from special standpipes, &c., making 593 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	104
New River, filtered (mean of 131 samples) ..	6
Thames, unfiltered (mean of 27 samples) ..	932
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 304 samples)	19
Ditto ditto .. .. highest	372
Ditto ditto .. .. lowest	0
River Lea, unfiltered (mean of 27 samples) ..	283
River Lea, from the East London Company's clear-water wells (mean of 41 samples) ..	6

The chief characteristic of the London waters during the month of May is the comparative microbic purity of the unfiltered waters of the New River, Lea, and Thames. During the month all the 557 samples examined from the clear-water wells showed that the filtration was being properly conducted, and was highly efficient as regards the elimination of microbes, with the exception that one of the Companies' wells on one occasion showed a higher number than 300 microbes. This arose from a purely local cause in one of the filters. It was at once remedied, and the action of the filter returned to its normal state of efficiency.

We are, Sir,
Your obedient Servants,
William Crookes.
James Dewar.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 7th, 1900.

Professor Thorpe, C.B., F.R.S., President, in the Chair.

(Concluded from p. 255).

82. "*The Ultra-violet Absorption Spectra of some Closed Chain Carbon Compounds.* II. *Dimethylpyrazine, Hexamethylene, and Tetrahydrobenzene.*" By W. N. Hartley, F.R.S., and Jas. J. Dobbie, D.Sc., M.A.

It was shown by the authors that dimethylpyrazine, a substance representing a group not hitherto examined spectroscopically, possesses a banded spectrum, the absorption band being both wider and more persistent than that of pyridine. This result was anticipated from the analogy of the constitution of this substance with that of benzene and pyridine. It affords further evidence of the intimate connection between the phenomenon of selective absorption and the benzenoid structure.

The result is further interesting in connection with the question of the constitution of cyanuric acid (*Trans.*, 1882, xli., 48). While pyridine contains the group ·C:N· once in the benzene structure, dimethylpyrazine contains it twice, and the formula originally proposed for cyanuric acid contains it three times. Accordingly, if this formula were correct for cyanuric acid, we should expect that it would exhibit a powerful absorption band more intense than that of pyrazine. It has, however, been recently shown (Hartley, *Proc.*, 1899, xv., 46) that cyanuric acid has no absorption band.

Specimens of pure hexamethylene and tetrahydrobenzene, prepared by Professor Sydney Young and Miss Fortey, and kindly lent by them, were found to be much more highly diathinic than benzene, and showed no trace of selective absorption. This result confirms the conclusion, previously drawn from the examination of such substances as hexachlorobenzene and piperidine (*Phil. Trans.*, 1885, Part II.), that with reduction of the benzene ring the substances are no longer characterised by an absorption band, but by a continuous spectrum indicating a less compact form of linking within the nucleus of the carbon atoms or of the carbon and nitrogen.

83. "*A Study of the Absorption Spectra of o-Oxycarbanil and its Alkyl Derivatives in relation to Tautomerism.*" By W. N. Hartley, F.R.S., Jas. J. Dobbie, D.Sc., M.A., and Photios G. Paliatseas.

Two ethyl derivatives of *o*-oxycarbanil, $C_7H_5NO_2$, are known. One of these is prepared directly from *o* oxycarbanil by boiling it with equivalent quantities of ethyl iodide and alcoholic potash. This ether is considered to have the lactam constitution, because when heated with hydrochloric acid it takes up water and decomposes into carbon dioxide and the hydrochloride of ethyl-*o*-amidophenol. The other ether, which is obtained by the interaction of *o*-amido phenol and ethyl imido-carbonate, yields oxycarbanil on treatment with strong hydrochloric acid, and is therefore a lactime. The chemical evidence leaves it undecided whether *o*-oxycarbanil itself possesses the lactam or the lactime structure. On photographing the absorption spectra of the three compounds it was found that the spectra of *o*-oxycarbanil are almost identical with those of the lactam ether. The amount of general absorption is practically the same in both cases, and the absorption band, which is well marked and persistent, occupies the same position in the spectra of both substances. The spectra of the lactime ether show a smaller amount of general absorption and the absorption band is much less persistent. From the similarity between the spectra of *o*-oxycarbanil and the lactam ether, it is inferred that the parent substance in this case, as in the cases of isatin and carbostyril (*Trans.*, 1899, lxxv., 640), possesses in solution the ketonic structure, or—if the solution be a mixture of tautomeric modifications—that the ketonic modification predominates almost to the exclusion of the enolic.

84. "*Action of Formaldehyde on Amines of the Naphthalene Series.*" II. By G. T. Morgan, D.Sc.

By the action of formaldehyde on ethyl-β-naphthylamine, 2 : 2-*diethyldiamino*-1 : 1-*dinaphthylmethane*, melting at 197—198°, is obtained when the reaction is carried out in cold glacial acetic acid, but when the condensation is effected in hot alcoholic solutions in the presence of hydrogen chloride, it is accompanied by a *compound*, $C_{23}H_{19}NO$ (m. p. 157—158°).

2:2-*Dibenzoyldiethyl*-1 : 1-*dinaphthylmethane*, produced from the preceding diamine by the Schotten-Baumann reaction, melts at 196°.

1 : 1-*Tetramethyldiaminodinaphthylmethane*, prepared from dimethyl-α-naphthylamine, melts at 177°; unlike its benzenoid analogue, tetramethyldiaminodiphenylmethane, it does not yield a colour base on treatment with lead peroxide.

Further investigation of the methylene bases derived from 1-bromo-2-naphthylamine and 1-chloro-2-naphthylamine has shown that these substances cannot be formulated on the type CH_2:NR, as indicated in the preliminary note (*Proc.*, 1899, xv., 10), but have the constitution indicated by the formula $CH_2(NHR)_2$. They do not combine additively with anhydrous hydrogen cyanide, and when treated with phloroglucinol (Tollens's reagent, *Ber.*, 1899, xxxii., 2841), they are quantitatively decomposed, yielding 2 mols. of the parent base and 1 equivalent of methylene-phloroglucinol $(C_7H_6O_3)_x$. It follows from these results that the bromo- and chloro-derivatives must be regarded as *methylenedi-1-bromo-2-naphthylamine*,—

$$CH_2(NH\cdot C_{10}H_6Br)_2,$$

and *methylenedi-1-chloro-2-naphthylamine*,—

$$CH_2(NH\cdot C_{10}H_6Cl)_2$$

respectively.

85. "*The Bromination of Benzeneazophenol.*" II. By J. T. Hewitt and W. G. Aston.

When benzeneazophenol is brominated in a glacial acetic acid solution containing an excess of fused sodium acetate, an almost theoretical quantity of benzeneazo-orthodibromophenol is obtained. If, however, the sodium acetate be omitted, a tribromo-derivative is formed, having a constitution hitherto unknown. The same product is obtained by bromination in aqueous suspension. This substance has now been proved to be *parabromobenzeneazoorthodibromophenol*. The proof has been furnished by brominating parabromobenzeneazophenol in presence of sodium acetate, and benzeneazoorthodibromophenol in presence of sulphuric acid. The same substance was obtained in both cases, and is identical with that obtained by the direct action of bromine on benzeneazophenol as described above. The corrected melting-point of the substance is 148°, and it has been further characterised by conversion into derivatives.

Acetyl ester, m. p. 167°; *benzoyl ester*, m. p. 129°; *ethyl ether*, m. p. 125°.

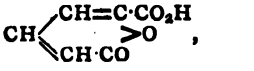
The isomeric *tribromobenzeneazophenol*, which was prepared from symmetrical tribromaniline for purposes of comparison, and contains the bromine entirely in the benzene nucleus, melts at 168·5°, its *acetyl* derivative at 105°, and its *benzoyl* derivative at 132°.

86. "*Condensation of Ethyl Crotonate with Ethyl Oxalate.*" By Arthur Lapworth.

A consideration of the points alluded to in a recent note (*Proc.*, 1900, xvi., 108), made it appear probable that ethyl crotonate and allied compounds would react in presence of sodium ethoxide with esters such as ethyl oxalate, and that substitution would occur, not in the α-position as usual, but in the γ-position, corresponding with the *meta*-position in benzoic acid, and to the position occupied by the hydrogen atoms in the methyl group of *o*- and *p*-nitrotoluene. This has been found to be the case.

Ethyl γ-oxalocrotonate, $EtO_2C{\cdot}CO{\cdot}CH_2{\cdot}CH{:}CH{\cdot}CO_2Et$, is easily prepared from ethyl crotonate and ethyl oxalate by the use of alcohol-free sodium ethoxide. It acts as a fairly strong acid, expelling carbon dioxide from carbonates, and yielding strongly coloured metallic derivatives. Its alcoholic solution is coloured brownish-black by ferric chloride, so that it exists, to some extent at least, in its "enolic" form. It forms nearly colourless crystals melting at 78—80°.

When ethyl γ-oxalocrotonate is hydrolysed by boiling with alkalis it does not give oxalic acid; with hydrochloric acid, *α-pyrone-α'-carboxylic acid*,—



is obtained. This separates from hydrochloric acid in colourless crystals melting at 227—228°.

The formation of ethyl γ-oxalocrotonate in the above manner recalls the observations of Henrich on ethyl glutaconate (*Ber.*, 1898, xxxi., 2103), but in the present case the $-CH_2$-group is not united to two unsaturated groups.

There are many facts bearing on "the negative nature of unsaturated groupings" which seem to point to the insufficiency of the view that the hydrogen atom in the grouping—

$$:CH{\cdot}A{:}B \text{ or } CH\lessdot^{A:B}_{C:D}$$

is of itself immediately replaceable, and it appears more likely that the position of attack is usually at a double linkage (compare Henrich, *Ber.*, 1900, xxxiii., 1437). In the above case, as in many similar ones, it appears not improbable that an intermediate compound derived from a tautomeric form—

$$\cdot CH{:}CH{\cdot}CH{:}C\lessdot^{OEt}_{OH},$$

is produced, and that subsequently, either by addition or isomeric change or by both, the γ-carbon atom becomes united with the substituting group (*Trans.*, 1898, lxxiii., 446).

The author proposes to extend the above observations, and to examine the capability of other $\alpha\beta$-unsaturated esters of taking part in this and similar condensations.

87. "*Researches in Silicon Compounds. VI. On Silicodiphenyldiimide and Silicotriphenylguanidine.*" By J. Emerson Reynolds, Sc.D., M.D., F.R.S.

The author has already described (*Trans.*, 1889, lv., 474) the well defined crystalline silicophenylamide, $Si(NHC_6H_5)_4$, the first of a new class of compounds in which silicon is exclusively united to nitrogen. It was there stated that when this compound was heated in a vacuum to its melting-point (137°) some aniline was given off and a mixture of silicon compounds formed, the constituents of which would be described later. The nature of these products was stated shortly afterwards to the British Association in 1893, but no details were then given. These are now described, before an account of more recent investigations is presented to the Society.

The primary interest attached to the examination of the changes effected by heating silicophenylamide is due to the probable production of silicon analogues of cyanogen compounds.

The study of the action of heat revealed the formation of the *diimide*, $Si(NPh)_2$, which exists in two modifications, one insoluble in benzene and the other soluble, but precipitated by ligroin in droplets which slowly change to the insoluble form. *Silicotriphenylguanidine*,—

$$SiNC_6H_5(NHC_6H_5)_2,$$

was isolated from the earlier products of the action of heat in small, short rhombic prisms, soluble in benzene. Like all other organic silicon compounds of this kind, it is decomposed by acids, as well as by water and alcohol.

The continued action of heat on the diimide gave rise to a compound represented by the formula $Si_2(NC_6H_5)_3$; this withstood a high temperature without charring, and when heated in dry ammonia left a residue which approximated in composition to $Si_2(NH)_2NC_6H_5$, but the decomposition could not be carried beyond this point without the separation of carbon.

88. "*Note on Bach's Hydrogen Tetroxide.*" By Henry E. Armstrong.

It is contended by A. Bach (*Ber.*, 1900, xxxiii., 1506) that the liquid obtained on mixing a solution of hydrogen peroxide with sulphuric acid probably contains hydrogen tetroxide. He bases this conclusion on the observation that when the amount of permanganate which the solution decolorises is contrasted with the amount of oxygen which is evolved, the latter is largely in excess of that which would be obtained if only hydrogen peroxide were present. Bach has obviously failed to take into account the existence of persulphuric acid in such a solution.

In all his experiments—both in those in which palladium hydrogen was oxidised and in those with peroxides—a large excess of normal sulphuric acid was added; the solution was then titrated with permanganate, and the oxygen which was given off was collected and measured. It is noteworthy that the liquid was shaken during several minutes (5—8) after the titration was completed in order to expel the gas. It is open to question whether, in these cases, some persulphuric acid was not formed (Lowry and West, p. 126) by peroxidation of the sulphuric acid, and whether the extra oxygen obtained by Bach was not derived from this.

Bach regards the fact that other dehydrating agents besides sulphuric acid—for example, hydrogen chloride—produce effects, when mixed with hydrogen peroxide, similar to those obtained on using Caro's reagent, as evidence in favour of the view that hydrogen peroxide is converted into tetroxide by dehydration,—

$$3H_2O_2 = H_2O_4 + 2H_2O.$$

It may be pointed out, however, that the effects produced

by Caro's reagent are merely such as it might be anticipated would be exercised by hydrogen peroxide acting in presence of a powerful dehydrating and hydrolysing agent, and it is the use of hydrogen peroxide under such conditions that characterises the novelty in procedure. It is not necessary to assume the existence of any novel specific agent in order to explain them. Caro's original observations stand on a different plane, as they served to establish a difference between the perdisulphates and salts of another acid, and for the first time made it possible to differentiate the acid products of the electrolysis of sulphuric acid.

PHYSICAL SOCIETY.

Ordinary Meeting, June 22nd, 1900.

Mr. T. H. BLAKESLEY, Vice-President, in the Chair.

A PAPER entitled "*Notes on Gas Thermometry*," by Dr. P. CHAPPUIS, was read by Dr. HARKER.

The author, having been led to recognise that hydrogen could not be used as a thermometric substance at high temperatures, on account of its action on the walls of the glass reservoirs, has had recourse to a constant volume nitrogen thermometer with an initial pressure slightly under 800 m.m. The value of the coefficient of expansion of nitrogen at constant volume is variable, diminishing up to 80° C. and then increasing slightly. In fact, nitrogen at 100° C. behaves like hydrogen at the ordinary temperatures, its compressibility being less than that required by Boyle's law. A table of corrections was therefore prepared. The readings of the constant volume nitrogen thermometer are too low, but the corrections are small, amounting to about 0·04° C. at the temperature of boiling sulphur. The mean result of the author's experiments for the boiling-point of sulphur is 445·2° under a pressure of 760 m.m. Callendar and Griffiths' result, obtained with a constant pressure air thermometer, is 444·53°. The difference is attributed to the joint action of several causes:—(1) The corrections for a constant pressure thermometer are about double those of a constant volume instrument; this correction applied to Callendar and Griffiths' result would raise it about 0·1°. (2) Callendar and Griffiths have used a value for the gas constant which is larger than that obtained by more recent experiments; adopting the latter value the boiling-point would be raised to 445°. (3) The divergence may be due to the expansion of the reservoir. The most accurate way of determining this is by the interference method of Fizeau. This method is used with small pieces of the material, and the author has employed it to determine the coefficient of expansion between 0° and 100°. Extrapolation to 450° might cause errors. The linear expansion has recently been determined by Bedford between 0° and 840°, by a comparator method. The homogeneity of porcelain is doubtful, especially when glazed, and the great differences occurring between the expansions obtained from the above methods is attributed to the change in form of the tube in Bedford's experiments, brought about by unequal thickness and want of homogeneity, and consequent unequal expansion. The author, therefore, adheres to his value of the boiling-point obtained from the expansion by the Fizeau method, whilst recognising the uncertainty attaching to the application of the coefficient of expansion of the reservoir over an interval four times as great as that over which it was determined.

A paper on "*A Comparison of Impure Platinum Thermometers*," by Mr. H. M. TORY, was read by Prof. CALLENDAR.

The object of this paper is to investigate the probable order of accuracy attainable in the determination of high temperatures by the use of ordinary commercial specimens of platinum wire. Five wires were compared from 400° to 1000° C. The fundamental coefficients of the wires varied within 40 per cent of the maximum value, but the temperatures observed by them—when calculated on the platinum scale by means of the ordinary simple formula—did not differ by more than 9° at 1000° C. Each wire was directly compared with a pure standard wire, the two being wound side by side in the same tube. Curves have been drawn with the platinum temperatures of the standard wire as abscissæ, and the differences between the temperatures indicated by the two wires compared as ordinates. These curves are all straight lines, within the limits of observation, and hence the determination of two constants is sufficient to enable us to compare an impure platinum thermometer with the standard, and therefore with the scale of the gas thermometer. The two constants can at once be obtained from observations at the boiling-point of sulphur and the freezing-point of silver, and thus a practical thermometric scale can be established, which between 0° and 1000° never differs by more than two or three degrees from the gas scale.

Prof. CALLENDAR said he was unable to agree with the correction to his observations suggested by M. P. Chappuis. He considered that the uncertainty in the coefficient of expansion of the gas was due to uncertain changes in the volume of the bulb, and to uncertainty in the coefficient of expansion of mercury. The fundamental coefficient of mercury was 0·00018153 according to Regnault, 0·00018216 according to the later reduction of Broch, and 0·00018256 according to experiments by Chappuis with a hard glass bulb. It made a difference of no less than 4 per cent in the fundamental coefficient of expansion of the glass, according as the original results of Regnault, or the value found by Chappuis assuming the linear expansion of the glass, were adopted. The importance of the changes in the volume of the bulb had been fully pointed out, and a method of taking approximate account of these changes had been explained in the paper on the boiling-point of sulphur in 1890. Unfortunately the glass employed was rather soft, and the changes of volume which occurred were too great to permit of the most accurate determination of the coefficient. The boiling-point when corrected for the smaller expansion of the bulb came out lower than 444·53. With regard to porcelain, Prof. Callendar did not consider it a good material, on account of the glaze. He did not think that the average coefficient of a tube or bulb over a large range of temperature could be inferred from a small and possibly asymmetric specimen. The results might be less inconsistent in the case of homogeneous and well-annealed metallic bulbs. The correction for the expansion of the bulb was, he believed, given by the expression $dt=(c+b\theta)\,t\,(t-100)$. He did not agree with M. P. Chappuis that the correction was independent of c, although the value of b was certainly most important at high temperatures. He also wished to take exception to the method adopted by Chappuis of calculating the correction of the nitrogen thermometer. According to Joule and Thomson the correction should be greater; according to other authorities it might be less. He hoped to discuss this in a future communication to the Society.

Mr. GLAZEBROOK said that, although he placed confidence in Chappuis' formula for a definite piece of porcelain between certain temperatures, he thought further and careful work was necessary before fixing on a formula for ordinary use.

Prof. CARHART said he would like to see a comparison made between the results of experiments with gas thermometers and those with platinum and platinum-rhodium couples.

Mr. ROSE-INNES expressed his interest in the behaviour of nitrogen about 100° C., as mentioned in M. Chappuis' paper.

Dr. LEHFELDT said the peculiarities of the nitrogen scale, between 70° and 80° might be explained by the reversal of the properties of nitrogen between 0° and 100°.

A paper on "*The Law of Cailletet and Mathias, and the Critical Density*," was read by Prof. S. Young.

The law of Cailletet and Mathias is very nearly, though in most cases not absolutely, true. It appears to be only strictly true when the ratio of the actual to the theoretical density at the critical point has the normal value 3·77. The curvature of the "diameter" is generally smaller the nearer this ratio approaches its normal value. The curvature is in nearly every case in opposite directions, according as this ratio is greater or less than 3·77. The curvature is generally so slight that the critical density may be calculated from the mean densities of liquid and saturated vapour at temperatures from about the boiling-point to within a few degrees of the critical point, with an error generally not exceeding 0·1 per cent. If, however, the critical density is calculated from the mean densities at low temperatures, the error may be considerable; in the case of normal decane it is between 5 and 6 per cent. The law does not, as a rule, hold good at all for substances the molecules of which differ in complexity in the gaseous and liquid states.

Mr. Rose-Innes said that in his paper the author had used the generalisations of van der Waals, although the author himself had shown that they were not strictly true.

Prof. Young said that the generalisations held in some cases, although they did not in others. In all cases they were approximately true, and it was therefore advisable to use them, and study the results as far as possible.

The Society then adjourned until next October.

NOTICES OF BOOKS.

Compulsory Licenses under the Patents Acts. By J. W. Gordon. Stevens and Sons, Lim., Chancery Lane.

Mr. Gordon has written a treatise on Clause 22 of the Patent Law of 1883, which should be of value to all classes of the community; to lawyers, as giving a clear account of the manner in which the Board of Trade is administering the novel powers conferred on it seventeen years ago; to patentees, as showing how the privileges which they claimed, and in some cases misused, are modified or abolished; and to the public at large, and especially manufacturers of patented goods, as showing the steps taken by the legislature to restore to them their ancient rights under the common law which they had, largely by their own supineness, allowed to be wrested from them.

Mr. Gordon, in explaining the privileges and obligations conferred on patentees by their grants, takes us back three hundred years, and shows us that, in the conditions then prevailing, the grant in the majority of cases served two purposes; the first was to enable the patentee to work his patent, the second was to secure to him a sufficient reward according to the merit of his invention. In early days the former was the all-important purpose, while so little was the latter considered that it was stipulated that should the working of the patent be to the detriment of the commonwealth, the grant would be cancelled. Of late years the former reason has, by virtue of changing circumstances, become obsolete, while patent owners have, as is natural, made full use of the latter.

When all the trades in the kingdom were governed by Chartered Guilds, and it was impossible for any person to practise his trade in any place where he was not a freeman, it was a necessary act on the part of the Crown for the general good of the community to confer "personal charters" or patents on inventors to enable them to work their inventions; but, lest they should use their privileged position to stifle an existing industry for their own personal gain, a drastic penalty clause was inserted, and, to make the purpose of the grant still clearer, it was expressly stated the reward, if any, gained by the inventor, was because he had introduced a new industry into the kingdom.

Since the decay of the Guilds, inventors and their backers have in many instances used their powers solely for their own profit, irrespective of the public good, whilst the penalty clause appears to have been forgotten.

In 1883 the Government passed an Act authorising the Board of Trade to compel a patentee to grant licenses to others to work his patent on terms to be decided by them, for either of three reasons duly set forth in Clause 22.

For various reasons this clause was not called into use for fifteen years; but in the last two years several applications for compulsory licenses have been made to the Board with varying success, and in the second part of his book Mr. Gordon discusses these cases, showing how various legal points were raised and disposed of, and why certain cases broke down, whilst others, which were superficially similar, succeeded.

This part of the book should be of assistance to lawyers, since it can hardly be doubted that the forms and procedures now being hammered out will serve as precedents to be followed in future cases.

The book is well written in language to be easily understood by the lay reader, though in many cases the punctuation is unorthodox; this is possibly due to the well-known antipathy between lawyers and all stops but periods. This slight blemish can be easily remedied in the next edition.

Outlines of Physical Chemistry. By A. Reychler. Translated by Dr. John McCrae. London and New York: Whittaker and Co.

The ground covered by this work is very extensive, and, as it is but a small volume, it is aptly labelled "Outlines."

Although there is no direct statement to that effect, the work appears to be based on a course of lectures delivered by Prof. Reychler at the University of Brussels, and would thus be of great value to students at that Institution; still the wisdom of adding it to the mass of technical literature already published in England is not obvious.

Prof. Reychler has treated his subject in four parts, with subdivisions.

In the first part he begins with stating the laws of chemical combination, leading to a discussion on gases and vapours under pressure, and by an easy transition to the methods of determining atomic weights, and concludes with a comparison of Mendeléeff's and L. Meyer's classification of the elements.

The second part treats of the transformation of matter through the solid, liquid, and gaseous states, with an examination of the phenomena of polarisation.

Thermo- and electro-chemistry are considered in the third part, and chemical mechanics in the fourth.

The author's views are in many cases not to be divined, for he sets forth opposing theories with the objections to each with such impartiality that one can only conclude that both are equally correct in his opinion.

A feature that might be more widely adopted is a complete index of author's names.

The illustrations are clear, and show all that is necessary without useless details.

The Tutorial Dynamics. By William Briggs, M.A., &c., and G. H. Bryan, Sc.D., F.R.S. London: Published by W. B. Clive.

This is one of a series of books written and published for that large and increasing class of students who study at home and correspond by post with their tutors. The authors have treated their subject in a very simple manner, the use of the calculus being altogether ignored, since they have found by experience that it is a dangerous weapon to put into the hands of men who are not thoroughly grounded in the elements of mechanics.

The volume covers the ground usually treated in such books, and does so in a satisfactory manner; it is as well, however, to point out that in Chapter XIV., on Motion down Inclined Planes, the inclination of "1 in n" is defined as the ratio of height to length, whereas engineers invariably use the ratio of height to base. The examples are numerous and well chosen. The addition of an index would be an improvement.

CORRESPONDENCE.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—If the Rev. G. F. M. Fielding will kindly refer to Perkin and Kipping's "Organic Chemistry," vol. 1, p. 16, he will find the following statement:—

"A few scraps of unglazed porcelain . . . prevent bumping."

Also, Gattermann, "Organic Chemistry," p. 30:—

". . . pieces of porcelain, powdered talc, . . . pieces of pumice bound with Pt wire" prevent bumping.

Also, Streatfeild's "Organic Chemistry," p. 10:—

"Two or three pieces of broken pipe-clay are added in order to prevent bumping."

Although pieces of broken tobacco-pipe are not mentioned, unglazed porcelain is very similar, and in my experience has always yielded good results.—I am, &c.,

CHEMICO.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—Mr. Fielding's suggestion, that in order to prevent bumping during distillation, small fragments of common tobacco pipes are very efficient, is one which *in principle* is pretty generally known. Tobacco piping itself may not be used, but unglazed earthenware (such as is used for drying chemicals) or other *porous* materials are extensively employed.

As Mr. Fielding (*re* his reply to "A Ghost of 200 Years") has only come across one suggestion, I may refer him to Gattermann's excellent little book on "Practical Methods of Organic Chemistry" (English translation, The Macmillan Co., 1896), where he will find some 18 pages devoted to distillation, and on page 30 the following substances recommended:—"Splinters of wood the size of a match, capillary tubes, bits of glass, pieces of porcelain, powdered talc, scraps of platinum foil or wire, small pieces of pumice stone, bound with platinum wire, also act satisfactorily."

As such a book as Gattermann is in most laboratories, Mr. Fielding need not be surprised when he gets such an answer as that from "A Ghost of 200 Years." His letters, however, furnish a reason why such letters should be published, letters which I thought were quite unnecessary.—I am, &c.,

W. R.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—If Mr. Fielding will refer to "Allen's Commercial Organic Analysis," vol. iv., p. 150, he will find the following:—". . . . and a few fragments of pumice or tobacco-pipe added to prevent bumping."—I am, &c.,

W. A. CATES.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 22, May 28, 1900.

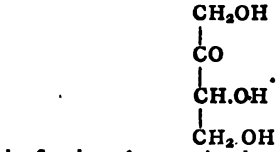
Formation of Nitric Acid in Combustions.—M. Berthelot.—The author continues his researches on the formation of nitric acid during combustions. He considers the case of the burning of sulphur in oxygen containing 8 per cent of nitrogen, under different pressures, and also in atmospheric air. The next series of experiments are performed on metals, to find what proportion of nitrogen becomes combined during the combustion of metals. The two metals used are iron and zinc.

Preparation, Properties, and Analysis of Thionyl Fluoride.—H. Moissan and P. Lebeau.—During the preliminary experiments, the authors found that when fluorine acts on sulphur in a glass vessel a mixture of fluorides and oxyfluorides of sulphur are formed, amongst which fluoride of thionyl can be recognised, SOF_2. This substance, after separation from the other fluorides, appears as a colourless gas, fuming in damp air, and with a disagreeable odour. Its boiling-point is about $-32°$, and density varies between 2·88 and 3·04, the theoretical number being 2·97. It is soluble in arsenic chloride, ether, essence of terebenthine, and benzine. The action of heat on this substance in the presence of glass allows of the determination of its composition by volume. Four volumes of SOF_2 giving four volumes of SO_2 and two volumes of SiF_4.

A Peroxide of Lithium.—M. de Forcrand.—The combustion of lithium in an atmosphere of oxygen gives mere traces of the peroxide; this body appearing to dissociate into the protoxide and oxygen. The author finds that, using the wet method of preparation, by allowing peroxide of hydrogen to act on a solution of lithia, a peroxide is formed. Two separate experiments were tried; the crystals separating out in the two cases having formulæ $Li_2O_2.H_2O_2+3·07H_2O$ and $Li_2O_2.H_2O_2+2·77H_2O$.

The Unknown Earths contained in Crude Samaria.—Eug. Demarçay.—The author previously announced that he had obtained from crude samaria a new element characterised by several spectrum lines, and having an atomic weight higher than that of samarium and lower than that of gadolinium. This substance he now obtains in a greater state of purity. The earth is white, slightly tinged with rose-pink; the salts being of a pale rose, and showing the following absorption bands in the nitric solution:—590, 570, 535, 525, 465, 395·5, 385·5, 380·5; none of these bands being strong. The characteristic spark spectrum lines are also given. The atomic weight of this element is about 151.

Hydrogenation of Erythrulose and Preparation of a New Erythrite, Dextro-erythrite.—Gabriel Bertrand.—The author has already shown that erythrite is rapidly oxidised by the bacteria of sorbose, and from it is obtained a syrup composed almost entirely of erythrulose. This new sugar contains four atoms of carbon, and has the formula—



By the fixation of one molecule of hydrogen, two stereoisomeric erythrites are formed: one inactive and identical with natural erythrite, the other optically active. This double transformation is effected by means of sodium amalgam.

Action of Cyanogen Chloride on Ethyl Acetonedicarbonate.—Juvénal Derôme.—One molecule of ethyl acetonedicarbonate is treated in the cold with a molecule of sodium ethylate, so as to obtain the mono-sodium derivative. This, in alcoholic solution, is then treated with a slow current of cyanogen chloride, the solution being kept cold. The reaction may be represented by the following equation:—

$$CO_2.C_2H_5—CHNa—CO—CH_2—CO_2C_2H_5 + CNCl = NaCl + CO_2C_2H_5—\underset{\displaystyle CN}{\underset{|}{CH}}—CO—CH_2—CO_2C_2H_5$$

This substance reacts again—

$$CO_2.C_2H_5—CHNa—CO—CH_2—CO_2C_2H_5 + CO_2C_2H_5—\underset{\displaystyle CN}{\underset{|}{CH}}—CO—CH_2—CO_2C_2H_5 = CO_2C_2H_5—CH_2—CO—CH_2—CO_2.C_2H_5 + CO_2C_2H_5—\underset{\displaystyle CN}{\underset{|}{CNa}}—CO—CH_2—CO_2C_2H_5.$$

This substance is extracted with ether, and on slow evaporation yields crystals of monocyanogen ethyl acetonedicarbonate.

Metallic Compounds of Diphenylcarbazone.—P. Cazeneuve.—Symmetric diphenylcarbazide, or phenylhydrazine urea, under certain oxidising influences, is transformed into a product called diphenylcarbazone. By allowing symmetric diphenylcarbazide to boil for about ten minutes with its weight of potash and twenty times its weight of alcohol at 93°, the derivative can be obtained in the form of an orange-red colouring matter, which is precipitated from a dilute solution by means of sulphuric acid. This substance has the formula—

$$CO\!<\!\begin{matrix}NM—NH.C_6H_5\\ N = N.C_6H_5\end{matrix}.$$

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 3.

New Method of Methylation.—Maurice Prud'homme.—Already inserted in full.

Mixed Anhydrides of the Acyclic and Cyclic Acids.—A. Béhal.—Already noticed.

Estimation of the Halogen Elements in Organic Compounds.—Amand Valeur.—Already inserted in full.

The presence of Mannocellulose in the Ligneous Tissue of Gymnosperm Plants.—Gabriel Bertrand.—The author has searched for mannocellulose in the ligneous tissue of a number of gymnosperm plants. All the species tried of the cycade and conifer families gave a fairly abundant precipitate of mannose hydrazone, sufficient even to recommend them as a good source of mannose. On the other hand, the *Ephedra distachya* gave very little, while the *Gnetum thea* and the *Welwitschia mirabilis* gave none at all.

On the Colouring Matter of Digitalis.—MM. Adrian and Trillat.—Already noticed.

MISCELLANEOUS.

The Spectaclemakers' Company and the Registration of Opticians.—It may be remembered that the Worshipful Company of Spectaclemakers, of which the Master is Sir Reginald Hanson, Bart. (the Senior Member for the City of London), recently inaugurated a scheme for the examination and certification of opticians, with a view to assuring to members of the public an adequate degree of scientific knowledge and skill on the part of those who make a business of fitting spectacles. The examiners are Prof. Silvanus P. Thompson, B.A., D.Sc., F.R.S., George Lindsay Johnson, M.A., M.D., F.R.C.S., W. H. E. Thornthwaite, F.R.A.S., and the Rev. J. Henry Smith, B.A. The following is a list of those who passed the fifth examination of the Company, held on the 28th, 29th, and 30th ult.:—F. Anderson, Elgin; H. B. De Brent, Beckenham; W. J. Bunt, London; R. A. Chadwick, Streatham; A. Charsley, London; J. R. Clarke, Newcastle; W. D. Eglinton, Lewisham; A. E. Esdaile, Liverpool; F. Fearnley, Leeds; J. J. Forbes, Stirling; S. H. Holroyd, Hull; E.W. Hovenden, Dulwich; H. A. Hughes, London; H. G. Husbands, Bristol; A. F. Ive, Worthing; J. W. Jones, Criccieth; R. P. Layfield, Liverpool; G. W. Lloyd, Sheffield; D. McFarlane, Manchester; C. Palmer, Gloucester; R. J. Scott, Waltham; W. Shelton, Stockport; G. Vogt, Kendal; J. B.Williams, Putney; W.Wood, Melbourne; A. V. Woodward, Derby. The total number of certified opticians is thus brought up to 296.

MEETINGS FOR THE WEEK.

Monday, July 2nd.—Royal Institution, 5. General Monthly Meeting.
Thursday, 5th.—Chemical, 8. The Nilson Memorial Lecture, by Prof. Otto Pettersson, of Stockholm.

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London:
CROSBY LOCKWOOD & SON, 7, Stationers' Hall Court, E.C.

UNIVERSITY OF BIRMINGHAM.
MATRICULATION EXAMINATION.

A Matriculation Examination will be held at the University during the week commencing Monday, September 17th, 1900. The last day for receiving applications for entry will be September 1st.—For further particulars of the subjects of examination and fees, &c., apply to the Registrar of the University.

An Assistant, experienced in Iron and Steel Analyses, is required by an Engineering Firm in the North of England.—Replies, stating age, experience, and salary required, to "Chemist," Chemical News Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Wanted, an Assistant willing to take charge of Stores, and capable of testing the same for purity and impurities.—Address, "Alpha," Chemical News Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Wanted, a Post as Laboratory Steward, or as Assistant in a Chemical Works Laboratory. Good references.—Address, E. Dancaster, Rus in Urbe, Green St, Jersey, C.I.

Wanted, for a period of six weeks, use of Laboratory in London. Analytical Chemist, who has private research work which will occupy that period, would be glad to receive particulars and terms for such an arrangement.—Address, F. R., Chemical News Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Youth, age 19, Assistant for three years in Public Analyst's Laboratory, desires Situation as Assistant or Pupil in Laboratory of Assayer or Metallurgist.—Address, J. S. D., Chemical News Office, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

ST. PAUL'S SCHOOL, LONDON.—An Examination for filling up about Sixteen Vacancies on the Foundation will be held on the 4th, 5th, 6th, 7th, and 10th September next.—For information apply to the Bursar of St. Paul's School, West Kensington.

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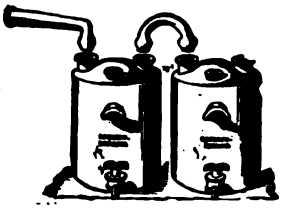

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